

# The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 69      Number 10

## CONTENTS

### PROCEEDINGS OF THE SOCIETY—

- THE APPLICATION OF IDENTIFICATION TINTS, WITH  
PARTICULAR REFERENCE TO ACETATE RAYON STAPLE  
(*H. C. Olpin and A. J. Wesson*) 357

- THE DYEING OF VISCOSE RAYON AT HIGH TEMPERATURES  
(*K. Butterworth*) 362

### COMMUNICATION—

- FURTHER OBSERVATIONS ON THE EFFECTS OF EVAPORATING  
WATER FROM COTTON CELLULOSE (*G. W. Madaras and H. A. Turner*) 371

- NOTES . . . . . 378

- NEW BOOKS AND PUBLICATIONS . . . . . 378

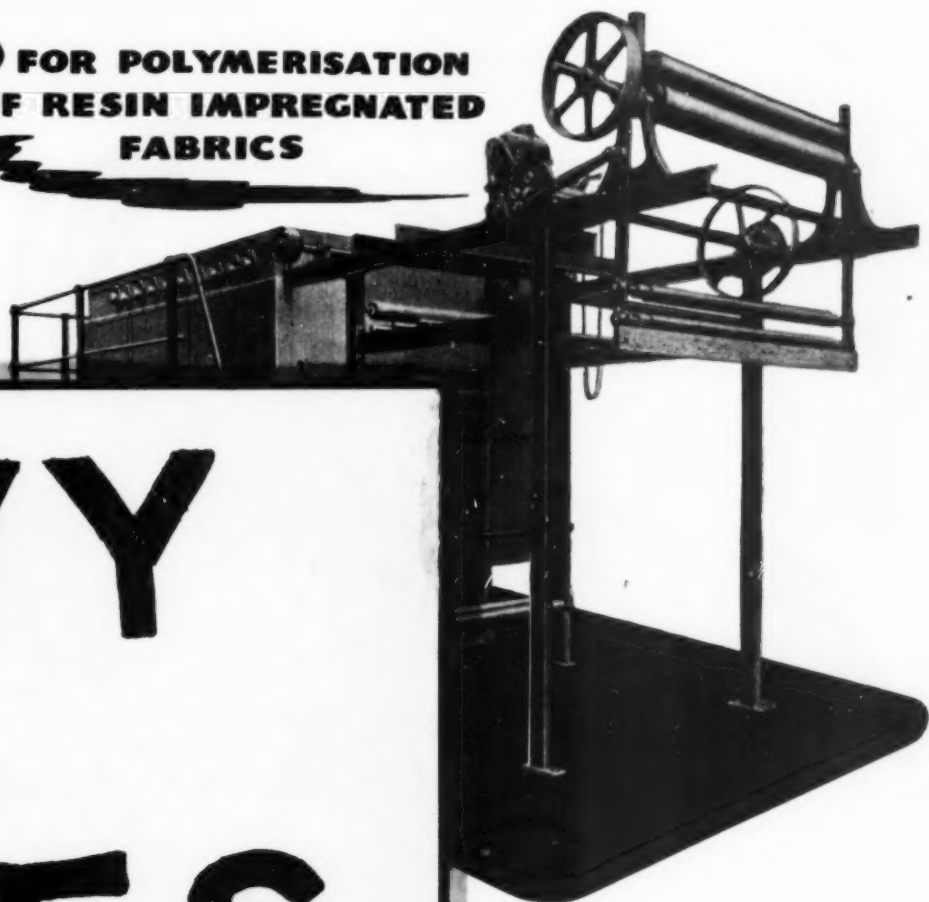
- ABSTRACTS . . . . . 382

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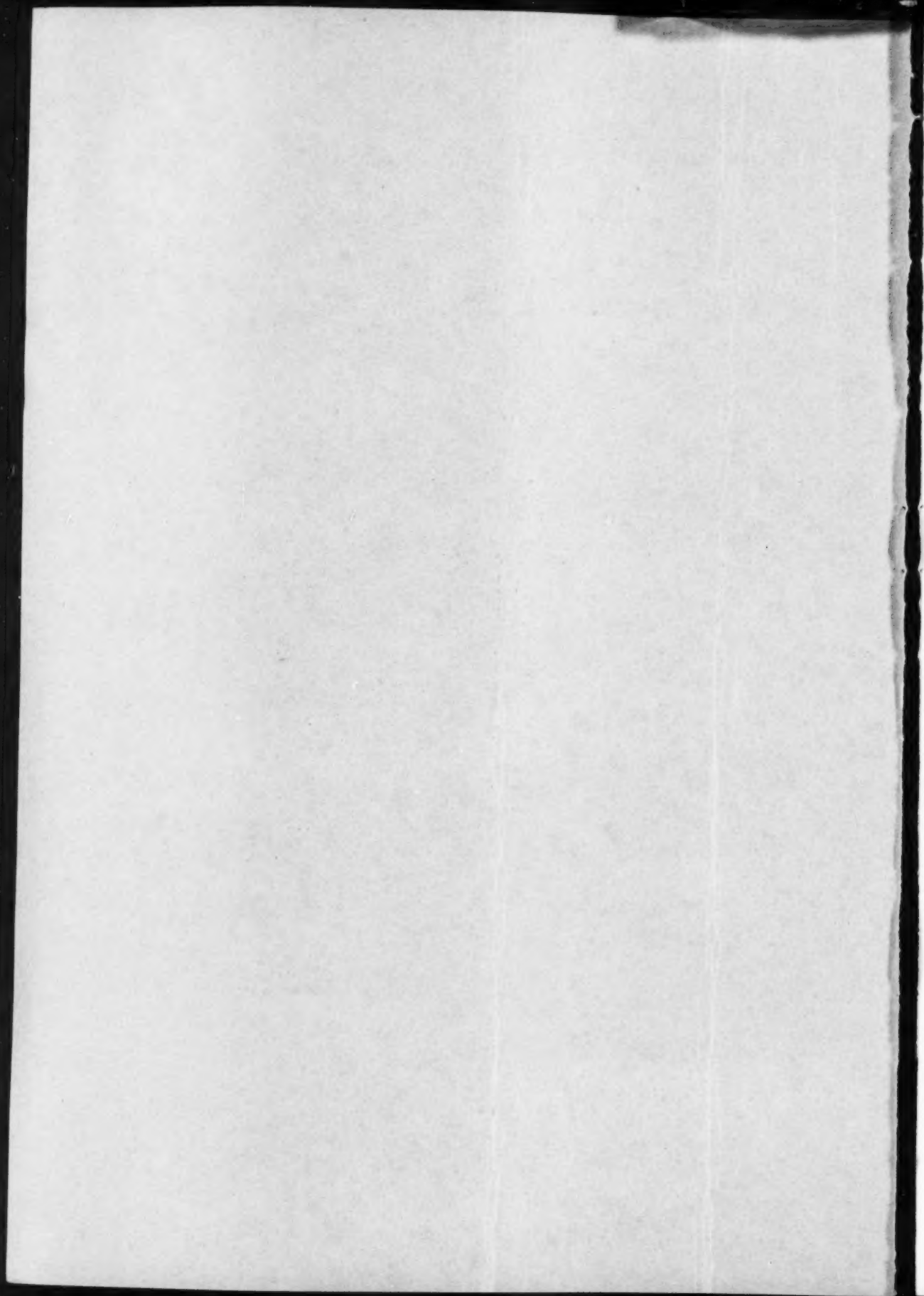
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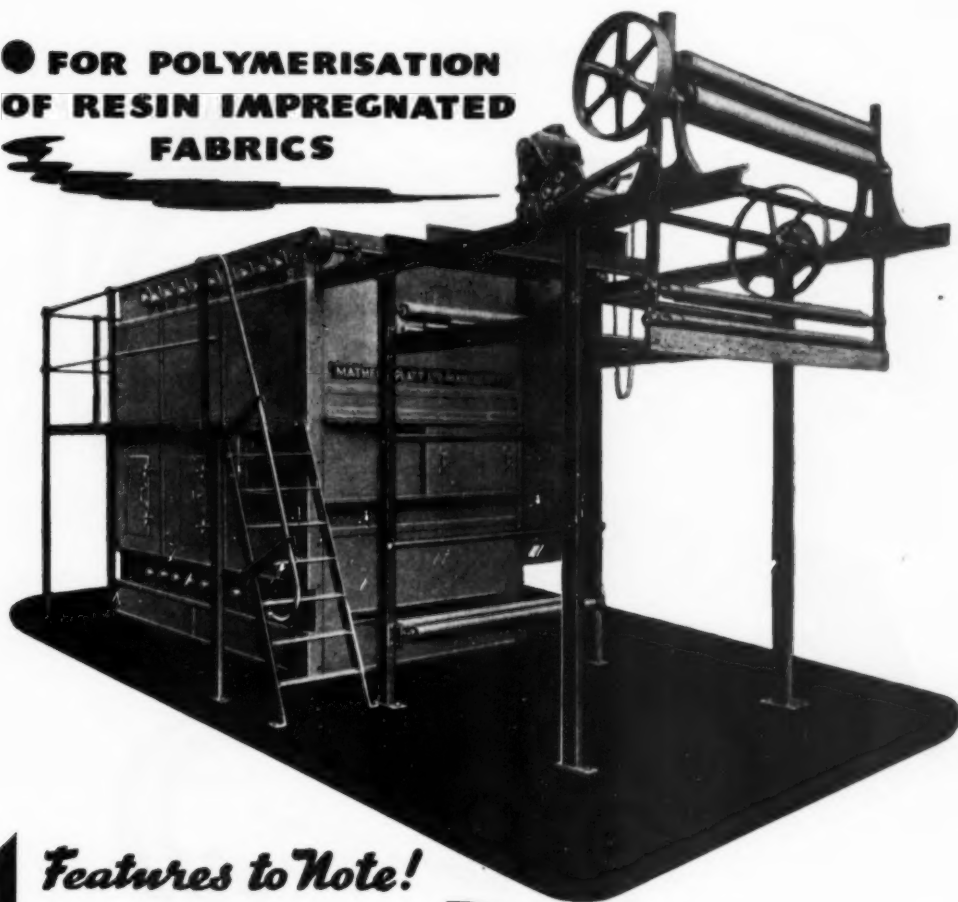
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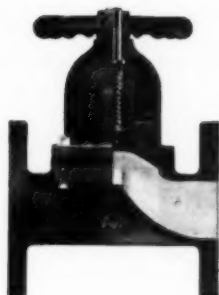
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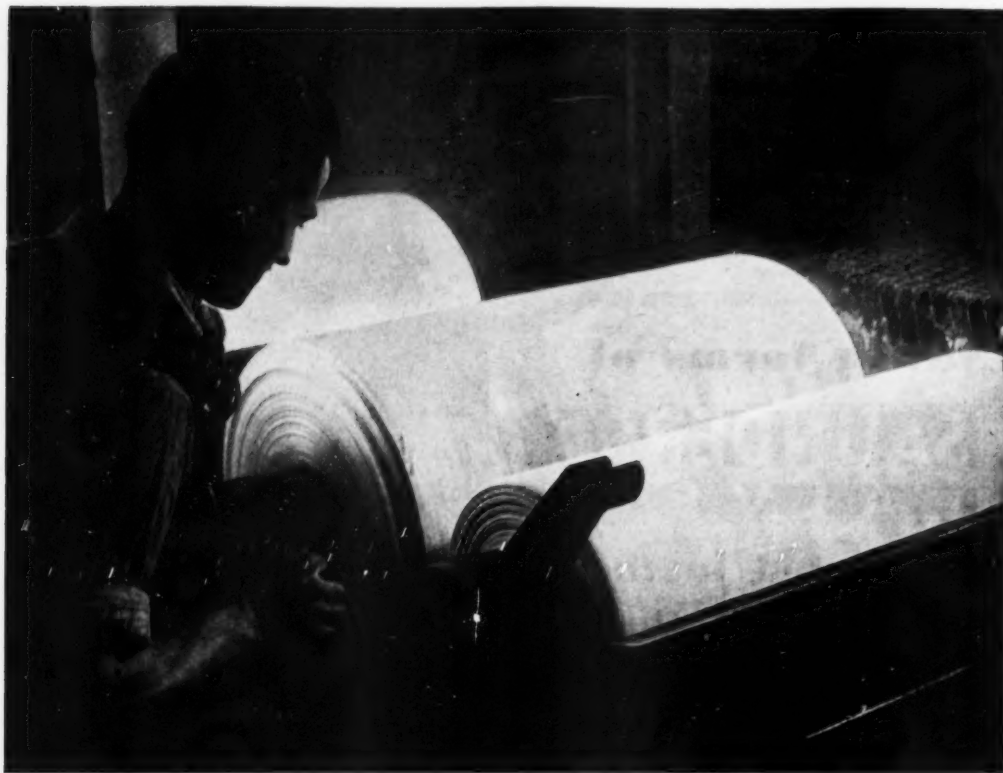


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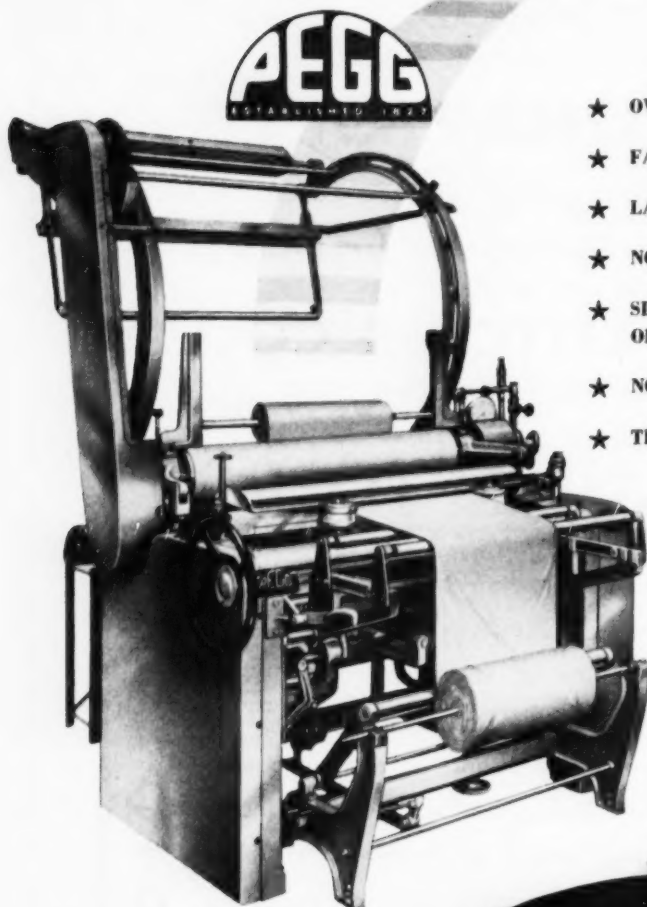
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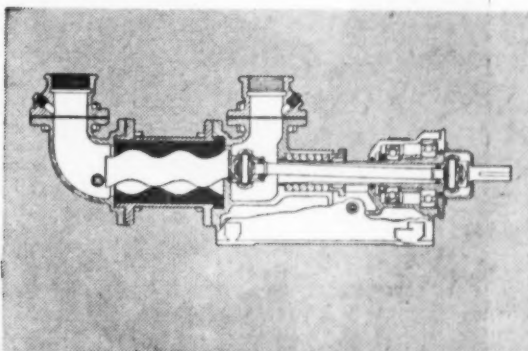
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## FORTHCOMING MEETINGS OF THE SOCIETY (see also page xxx)

### SCOTTISH SECTION

All Meetings in St. Enoch Hotel, Glasgow, at 7.0 p.m., unless otherwise stated

- 1953  
 Tuesday 27th Oct. Dr. G. H. Lister (Sandoz Products Ltd.). *The Migration Properties of Acid and Chrome Dyestuffs*  
 Tuesday 17th Nov. J. S. Ingham, Esq., M.Sc., F.R.I.C. (Marks and Spencer Ltd.). *Problems of Colour Fastness and Finish*  
 Thursday 10th Dec. Dr. J. F. Gaunt (Messrs. Patons & Baldwins Ltd.), Scottish Woollen Technical College, Galashiels. *A discussion on the Relative Merits of Different Methods of Applying Chrome Dyes*  
 Tuesday 15th Dec. Prof. P. D. Ritchie. *Links between the Textile and Plastics Industries*

### MIDLANDS SECTION

Except when stated otherwise, Lecture Meetings commence at 7.0 p.m.

- 1953  
 Tuesday 3rd Nov. D. N. Marvin, Esq., A.R.I.C., A.T.I. (Joint meeting with Hinckley Textile Society). *Some Properties of Terylene*. College of Further Education, Hinckley. 7.15 p.m.  
 Wednesday 18th Nov. H. R. Hadfield, Esq., M.Sc. *The Dyeing of Caprolactam Fibres (Perlon etc.)*. Midland Hotel, Derby  
 Saturday 21st Nov. LADIES EVENING — DINNER DANCE, Black Boy Hotel, Nottingham  
 Wednesday 2nd Dec. R. W. Speke, Esq., B.Sc. *A Study of Variables in Padding*. Black Boy Hotel, Nottingham  
 1954  
 Wednesday 20th Jan. J. Payne, Esq., B.Sc. *Silicon Chemicals in Textile Finishing*. College of Technology, Leicester  
 Wednesday 24th Feb. Douglas Haigh, Esq., A.T.I. *Some recent researches in the Dyeing of Felt*. Masonic Hall, Nuneaton  
 Thursday 4th March R. F. Wilson, Esq. (B.C.C.) (Joint meeting with the Nottingham Textile Society). *Colour*. Gas Theatre, Nottingham  
 Wednesday 17th March G. H. Lister, Esq., B.Sc., Ph.D. *The Migration Properties of Acid and Chrome Dyes*. Victoria Station Hotel, Nottingham  
 Friday 26th March MIDLANDS SECTION DINNER. King's Head Hotel, Loughborough  
 Wednesday 7th April ANNUAL GENERAL MEETING OF SECTION. J. A. Potter, Esq., M.A. *Dyeing and Finishing Synthetic Fibres in the Hosiery Industry*. King's Head Hotel, Loughborough

### LONDON SECTION

All Meetings will be held at the Royal Society, Burlington House, Piccadilly, London W.1, at 6.0 p.m. unless otherwise stated

- 1953  
 Friday 6th Nov. Messrs. B. Kramrisch and A. Bratt (Clayton Aniline Co. Ltd.). *Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons*  
 Thursday 3rd Dec. Alec B. Hunter (Warner & Sons Ltd.). *Design and Colour*

### LONDON SECTION (continued)

- 1954  
 Thursday 7th Jan. Dr. G. T. Douglas (I.C.I. Ltd.). *The Dyeing of Synthetic Fibres*  
 Friday 5th Feb. C. P. Tattersfield (Courtaulds Ltd.). *Dyeing and Finishing in relation to Use-Development Work*  
 Friday 5th March *The London Lecture*. Subject, Speaker and venue to be given

### MANCHESTER SECTION

Unless otherwise stated, meetings take place at the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

- 1953  
 Friday 20th Nov. LADIES' EVENING *How do I know what to buy?* Commences at 7.0 p.m. Details will be announced later  
 Friday 18th Dec. Details to be announced later  
 1954  
 Friday 15th Jan. S. Blackburn Esq., and M. R. Fox, Esq. (I.C.I. Ltd., Dyestuffs Divn.). *The Behaviour of Leuco Vat Dyes above 100°C.*

### WEST RIDING SECTION

Meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

- 1953  
 Thursday 29th Oct. Sir Ernest Goodale, C.B.E., M.C. *Ancient and Modern Textile Production in London and the Home Counties*

### NORTHERN IRELAND SECTION

All Meetings with the exceptions of "Ladies' Evening" and "du Pont Story" to be held in Royal Avenue Hotel, Belfast, at 7.30 p.m.

- 1953  
 Wednesday 28th Oct. LADIES' EVENING, to be held in Thompson's Restaurant, Belfast, at 7.30 p.m.  
 Friday 6th Nov. S. J. Hill, Esq. (United Chrometanners Ltd.). *Production and Dyeing of Leather*. Two films will be shown *Romance of Leather and Hide to Sole*  
 Friday 4th Dec. J. V. Summersgill, Esq. (Geigy Co. Ltd.). *Some Aspects of the Chemistry and Textile Uses of Sequestering Agents*  
 Thursday 10th Dec. *du Pont Story*. To be held in Thompson's Restaurant, Belfast, at 7.30 p.m. This is a colour/sound film. (Joint Meeting with Textile Institute)  
 1954  
 Friday 8th Jan. C. P. Tattersfield, Esq. (Courtaulds Ltd.). *Developing New Uses for Rayon*. (Joint meeting with Textile Institute)  
 Friday 5th Feb. W. Ford-Kirkpatrick, Esq. *Putting the Colour into Photography*  
 Friday 5th March J. Potter, Esq. (Clayton Aniline Co. Ltd.). *Special Finishes for Textiles and Their Evaluation*. (Joint meeting with Foremen Dyers Guild)  
 Late March or early April ANNUAL GENERAL MEETING AND DINNER. (Details later)

## MEMBERS' CHANGES OF ADDRESS

- Asbhy, W. L., formerly of Doncaster, to Highleigh, Whaley Lane, Whaley Bridge, near Stockport
- Bali, Yervant, formerly of Manchester, to 32 Baron Empain Avenue, Heliopolis, Egypt
- Beeley, E., formerly of Glastonbury, to Rock Cottage, Littleton, Somerton, Somerset
- Bielawski, F. W., formerly of c/o Turnbull's Ltd., to "Wilton Grove", Victoria Road, Hawick
- Broadhurst, R., formerly of 39 Caledonian Road, Leeds 2, to 89 Caledonian Road, Leeds 2
- Butterworth, J. F. D., formerly of Oldham, to "Rostherne", 1 Burlington Drive, Davenport, Stockport, Cheshire
- Cieslar, G., formerly of Nottingham, to 7 Merton Road, Bradford 7
- Cockhill, G. P., formerly of Gibbon Street, to Townley Chemical Co. Ltd., Gorton Lane, Manchester 12
- Desai, Dr. N. F., formerly of Shri Ram Institute, Delhi 8, to Imperial Mahal, Khodadad Circle, Dadar, Bombay, 14, India
- Dixie, J. A., formerly of Leeds 2, to c/o The Dominion Laboratory, Sydney Street, Wellington, New Zealand
- Gale, R. A., formerly of New Basford, to 135 Middleton Boulevard, Nottingham
- Glaser, R. D., formerly of Hasenrainstr 29, to Hasenrainstr 13, Binningen, (Baselland), Switzerland
- Graham, J. G., formerly of Crumpsall, Manchester, to 7 Home Drive, Alkrington, Middleton, Lancs.
- Hallows, H. B., formerly of 19 Fauvel Road, Glossop, to 43 Norfolk Street, Glossop, Derbyshire
- Heaton, R., formerly of Plumstead, to 30 Woolwich Common, Woolwich, S.E.18
- Hesse, L. M., formerly of Old Coulsdon, Surrey, to 12 Christchurch Place, Christchurch Mount, Epsom, Surrey
- Holt, J., formerly of Marple, Cheshire, to "Brookside", 40 Starling Road, Radcliffe, Lancs.
- Hovland, Hans, formerly of 4 Melbourne Place, to 14 Rawson Road, Bradford
- Inman, E. R., formerly of Horsforth, Leeds, to James Anderson & Co. (Colours) Ltd., Portland Road, Hawkhead, Paisley, Renfrewshire
- Jabr, S. U., formerly of 71 Whitworth Street, to 9 Dalston Drive, Didsbury Park, Manchester 20
- Lawman, D. K., formerly of Leeds, to 2 Windsor Drive, Timperley, Cheshire
- Lewis, G., formerly of 46 Wellington Street, to 96 Wellington Road, Coppice, Oldham, Lancs.
- Marsden, J., formerly of Prestwich, to "Rowlands", 7 Cranford Avenue, Whitefield, Lancs.
- Newsome, G., formerly of Thornhill, Dewsbury, to Croft House, Scopoley Lane, Whitley, Dewsbury
- Nunes, L. F. M., formerly of Eagley Lane, Eccleshill, to Mount Royd Hotel, Manningham Lane, Bradford
- Nuttall, H., formerly of 96 Compstall Road, to "Fourways", Chadkirk Road, Romiley, Cheshire
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- Porter, J. C., formerly of China, to 22 Whilby Avenue, Fartown, Huddersfield
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- Swidzinski, G. B., formerly of 40 Delannays Road, to 18 Brookfield Road, Higher Crumpsall, Manchester
- Thornton, G. L., formerly of Leicester, to c/o Messrs. Imperial Chemical Industries Ltd., Blackley, Manchester 9
- Trotman, G. H., formerly of Holmrook, to Golf Thwaite, Seascale, Cumberland
- Twitchitt, H. J., formerly of Derby, to 9 Broadoak Road, Bramhall, Cheshire
- Waldron, C. A., formerly of Renfrewshire, to 21 Wellport Terrace, Neilston, Glasgow
- White, M. K., formerly of Heaton, Bradford, to 41 Whetley Hill, Bradford, Yorks.
- Whiteley, C. E., formerly of Halifax, to 73 Bankside Lane, Bacup, Lancs.

## ADDRESSES WANTED

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- Crummett, A., formerly of 14 St. Austell Road, Manchester 16
- Evelbauer, H. L., formerly of Freidrichsster F., Marburg (16), Hessen, U.S. Zone, Germany
- Falconer, J. S., formerly of c/o Campbell, "Grasmere", Drymen Road, Balloch, Dumbartonshire
- Gaukroger, G. A., formerly of 28 Akeds Road, Halifax, Yorks.
- Kazan, R. E., formerly of c/o Howards & Sons Ltd., 815 Castle Buildings, 1416 Stanley Street, Montreal, P.Q., Canada
- Midgley, F. B., formerly of 27 Scholes Lane, Scholes, near Cleckheaton
- Molloy, G. Jnr., formerly of 4 Mount Prospect Park, Belfast, Northern Ireland
- Nayar, K. N. S., formerly of c/o U.T.M. Ltd., Anchor Mills, Paisley, Scotland
- Southwell, G., formerly of Hillcrest House, Scotchman Lane, Batley, Yorks.
- Sykes, J. H., formerly of 75 Hill Crescent, Howden Clough, Birstall, near Leeds
- Taylor, F. G., formerly of 4 Nevanthor Road, Leicester

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## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1953 and pages 229-232 of the July 1953 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

- The Textile Trade and the Newer Fibres *F. H. Clayton*  
The Heat Setting of Terylene Polyester Filament Fabrics in relation to Dyeing and Finishing *D. N. Marvin*  
Studies in the Printing of Terylene Polyester Fibre *A. G. H. Michie*  
The Uses of Microscopy in Textile Dyeing and Finishing *G. G. Taylor and J. C. Brown*  
The Dyeing of Nylon Yarn in Package Form *W. S. Willson*

### COMMUNICATIONS

- The Reaction between Wool and Nickelammonium Hydroxide.  
II—The Supercontraction of Wool Fibres in Solutions of Nickelammonium Hydroxide *J. W. Bell and C. S. Whewell*  
Studies in the Fundamental Processes of Textile Printing.  
VI—The Transfer of Disperse and Water-soluble Dyes to Nylon during Steaming *E. H. Daruwalla and H. A. Turner*  
Absorptometric Colorimetry in the Textile Laboratory *C. H. Giles and J. S. Shaw*  
Observations on Some Commercial Colour Matching Lamps *J. C. Guthrie and P. H. Oliver*  
The Effect of Dyeing on the Sorption of Water Vapour by Wool *P. Larose*  
Some Experiments on the Over-dyeing of Nylon *C. D. O'Brien and R. H. Peters*  
The Effect on Wool of Boiling in Aqueous Solutions.  
I—Water and Solutions of Sodium Sulphate at pH 1.5-9.0 *R. V. Peryman*



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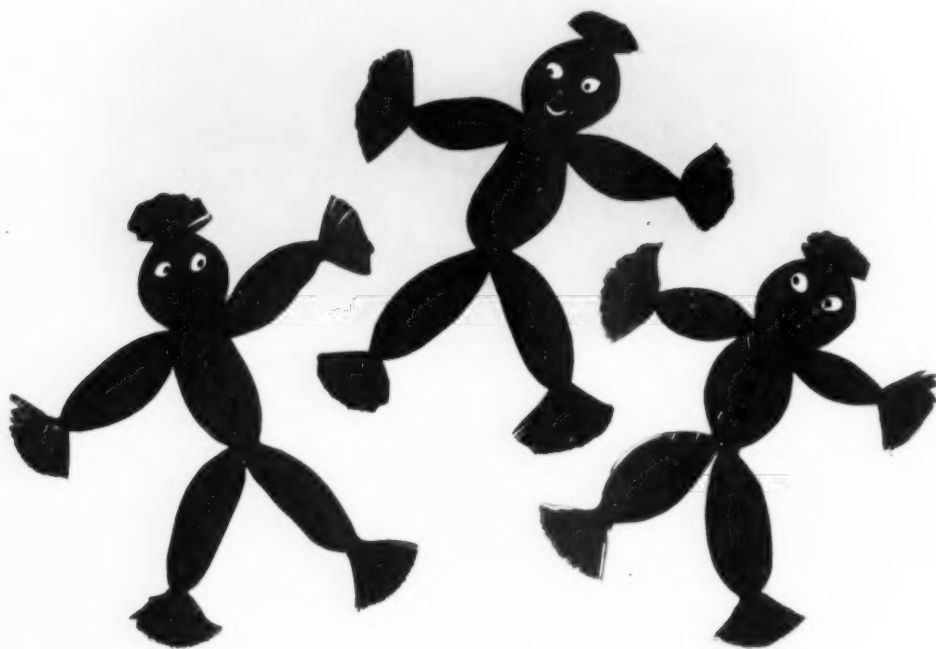
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# THE JOURNAL

OF THE

## Society of Dyers and Colourists

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Volume 69 Number 10

OCTOBER 1953

Issued Monthly

### Proceedings of the Society

#### The Application of Identification Tints, with particular reference to Acetate Rayon Staple

H. C. OLPIN and A. J. WESSON

*Meeting of the West Riding Section held at the Victoria Hotel, Bradford, on 13th November 1952,  
Mr. S. L. Peel in the chair*

##### Introductory

In the textile industry rapid and accurate identification of yarns or textile materials is essential, and the simplest and surest way to achieve this is to colour them according to a strict code. Different fibres, different forms of the same fibre such as different deniers and staple lengths, different classes of waste, and so on, come under consideration. For example, the sighting colours which differentiate between S and Z twists and between the different ends in a warp are of great importance in avoiding elementary weaving and knitting faults. The advent of man-made fibres has added urgency to the problems of fibre identification, as those who have seen the devastating effect of a single "rogue" end in a fabric, or a minute percentage of a "rogue" fibre in staple yarn, can testify.

The best solution to these problems is undoubtedly the application of colouring matters which may be readily and completely removed after they have served their purpose. But the subject of fugitive tinting is fraught with difficulties, and very few people have ventured to make authoritative statements on the matter, particularly where wool is concerned, unless the following passage from a pre-war publication of a well known foreign combine<sup>1</sup> can be accorded that dignity: "... there are no dyestuffs available which can be removed entirely by piece washing in every instance". The authors then proceed to take the view that what cannot be cured must be endured; in other words, a tint which cannot be removed must be utilised, and manufacturer and customer are advised to come to an understanding. Such advice is not very encouraging when it is considered against the desiderata of an identification tint, viz. that it must be readily removed or destroyed by washing or other simple means, that it must pass through all subsequent manufacturing

processes without being fixed, and that it must not transfer to and be fixed on other fibres which may be present.

This overriding consideration of ease of removal must necessarily determine the type of colouring matter selected for any particular purpose. Water is cheap, and the textiles will sooner or later come into contact with it, so water-soluble dyes have always been first choice. But, as we shall see later, they have their limitations and cannot be used indiscriminately.

In this survey it is not possible to consider every aspect of tinting on all fibres, and for brevity we propose to confine our remarks to identification tinting of acetate rayon staple, wool, and viscose rayon staple in relation to each other. The acetate staple we have used in our investigations is that marketed by British Celanese Ltd. under the registered trade mark *Celafibre*.

##### Colouring Matters used for Tinting

The materials available for tinting may be considered in three sections, viz. pigments, oil-soluble dyes, and water-soluble dyes, the latter being by far the most important.

##### PIGMENTS

Pigments have been used in the past, but they are all subject to one disadvantage, namely that they are in the nature of "soils", and it is extremely difficult to remove all "soil" from textile materials. They may be mechanically trapped in the yarn, particularly if it is of high twist, and may also be inadvertently fixed by chemical means, for example by reduction if they are vat dyes applied in a slightly thickened medium. A further type of pigment, represented by ultramarine, which is available in extremely finely divided form, is readily destroyed by acid; in spite of this, however, it has been known to cause trouble, owing to the

white or near white decomposition products being themselves mechanically held by the fibre or fabric. Abrasion is another potential source of trouble with pigments.

#### OIL-SOLUBLE DYES

Oil-soluble dyes dissolved in the spinning oils used in textile processing have met with only limited success, and are quite impracticable when only a very small percentage of oil is applied. When considerable quantities of oleine are used, as in the woollen industry, the chances of obtaining adequate distinctive coloration and of completely dispersing and removing such dyes later are much higher. Nevertheless they are little used, and would be valueless on Celafibre because of their staining propensities.

#### WATER-SOLUBLE DYES

We therefore turn to the water-soluble dyes, to consider which of those available are so lacking in affinity for one fibre or another that they can readily be removed at the appropriate time.

It is comparatively easy to apply aqueous solutions of selected soluble tints to most fibres, dry carefully, and remove them completely by a light scour. If this were the only consideration there would be few tinting problems on textile fibres today; but, whilst this very modest requirement is often the only one, it is at least as often the case that the tinted fibres have to undergo a whole series of manufacturing hazards, which in the past have made it quite impossible for dye manufacturers to provide any range of tints which will satisfy all requirements. They have therefore preferred to leave the various branches of the textile trade to work out their own salvation.

We will now consider the three main groups of water-soluble dyes—the basic, acid, and direct cotton dyes—in relation to the three fibres to which we are limiting our observations.

##### (a) Basic Dyes

Basic dyes have affinity for both wool and Celafibre. They are used on wool in a variety of tints, and are satisfactory for all-wool goods owing to their ease of removal by acid treatments. But since they have affinity for Celafibre under conditions favourable to their removal from wool, they are unsuitable for use on either component of a Celafibre-wool blend.

##### (b) Acid Dyes

This is a very extensive class, including the simpler sulphonated azo and sulphonated basic dyes, which have the virtues of brilliance of shade and high colour value. Generally speaking, these dyes have good affinity for wool, but none for either Celafibre or viscose rayon staple. Selected members are fugitive on both the latter fibres, and have been used for tinting them. Celafibre so tinted can be blended with either wool or viscose staple and processed to the finished goods. The tint is then readily removed from the Celafibre in a mildly alkaline scour bath, and does not stain either of the other fibres present.

The acid dyes still leave much to be desired, however. Many can be fixed on Celafibre in circumstances in which the dye may enter the fibre while the latter is in a moist, incipiently swollen state. They are then very resistant to subsequent scouring. This type of fixation may occur accidentally, owing to water spotting, or when the goods are left to condition on a damp floor, a practice which is common in the worsted industry, and to which Celafibre-wool cops are likely to be subjected.

Acid dyes can also be transferred completely from Celafibre to wool by treatment in a warm humid atmosphere, often given to set the fibre and prevent "snarling".

Should an alkali-sensitive triphenylmethane dye have been used, however, such fixation is often obscured because the mildly alkaline scour bath decolourises the dye by formation of the benzhydrol and reappears during dyeing.

##### (c) Direct Cotton Dyes

These have in the past received scant consideration for tinting purposes, owing to their affinity for viscose staple, and the propensity to stain or even to dye wool. At first sight, they would not appear to be very promising for tinting Celafibre destined for blending with either of these fibres, but recent work has led us to modify this view, and to suggest them as a solution to the problem of tinting Celafibre.

Any worthwhile solution to tinting problems must be of wide application, since it would be quite impracticable for manufacturers of staple fibre to offer a sufficiently wide range of tints to meet all requirements. One essential feature of any tinting material must be its covering power, which is very good on Celafibre with many of the acid dyes. Increasing the degree of sulphonation to increase the electronegative charge on the dye anion certainly reduces the possibility of incipient fixation on Celafibre, but introduces the further objection that bright shades are no longer given. Amaranth B (C.I. 184) is completely fugitive on Celafibre, but gives a very poor tint indeed.

#### CETYLPIRIDINIUM BROMIDE COMPLEXES

While we were examining a number of spreaders, such as gelatin<sup>2</sup>, ethylene oxide condensation products, and the like, to overcome this difficulty, it occurred to us that the cation-active quaternary ammonium compounds such as cetylpyridinium bromide might be useful<sup>3</sup>. This latter compound gives precipitates with acid and direct dyes which are soluble in excess of the quaternary compound to give complexes which *prima facie* would be very unlikely to enter the fibre themselves or to permit the tinting dye to do so.

Most acid dyes, of which the following are a selection—

	C.I. No.
Kiton Red G	31
Coomassie Acid Blue RLS	208
Xylene Fast Green B	735
Naphthalene Fast Orange 2G (ICI)	—
Acid Violet 4BNS	698

give lakes which are soluble in excess of the quaternary salt, and are fugitive on Celafibre even

after "swealing" and steaming treatments. After many laboratory trials we proposed proceeding to a larger scale. Difficulties arose almost immediately, however, the most serious objection being that most textile processing is carried out in anion-active systems with which the cetylpyridinium bromide is not compatible. We did not feel equal to the task of weaning the textile industry from anion-active to cation-active systems, and trials never proceeded beyond the exploratory stage.

#### POLYVINYLPIRROLIDONE COMPLEXES

Polyvinylpyrrolidone has recently been introduced by the Badische Anilin- und Soda-Fabrik under the trade name of *Albigen A*, a liquid containing about 30% of total solids. It is slightly yellowish and of medium viscosity, and is described as possessing pronounced dye-binding properties for leuco vat dyes, sulphur dyes, and direct cotton dyes. It is claimed to have a higher affinity for the dye than the cellulose fibre itself, and has been called a "liquid fibre" by the makers<sup>4</sup>. These properties were confirmed in the course of laboratory work, and its potentialities as a vehicle for fugitive tinting were visualised and investigated<sup>5</sup>.

In the proportion of 4-6 : 1 on the weight of dye, *Albigen A* substantially inhibits dyeing of a number of direct cotton dyes on cellulose, the extent varying with the individual dye. Thus Chlorantine Fast Blue 3GLL (CAC) is inhibited from dyeing, but Chlorazol Blue B (ICI) still dyes appreciably, as also do a number of other direct cotton dyes. This point is of particular importance, since much Celafibre is blended with viscose staple, the 50 : 50 mixture being very popular, and it must receive due consideration when a solution is sought for Celafibre-wool.

It is noteworthy that polyvinylpyrrolidone does not inhibit fixation of acid dyes on wool, nor does it entirely eliminate the possibility of their inadvertent fixation on Celafibre. Polyvinylpyrrolidone-acid dye complexes, therefore, are not of the same interest to us as direct cotton dye complexes.

From a large number of dyes examined to formulate a range, the following were chosen for further investigation—

- Durazol Fast Orange 2GS (ICI)
- Durazol Fast Blue 4RS (ICI)
- Chlorazol Fast Pink BKS (ICI)
- Chlorantine Fast Blue 3GLL (CAC)
- Chlorantine Fast Blue 4GL (CAC)
- Chlorantine Fast Green FGLL (CAC)
- Solophenyl Blue Green BL (Gy)
- Solar Green 5GL (S).

Two of these dyes were selected for special investigation in view of their high affinity for *Albigen A*, and we will now follow them through the various manufacturing processes to the finished product.

#### Pilot Trials and Fugitivity Tests

(1) Two batches of Celafibre-viscose staple (50 : 50) blend were spun on normal cotton-spinning machinery according to the following procedure—

The Celafibre component (2½ in. staple, 2½ filament denier, dull) was sprayed at the hopper feeder with 2½% of its weight of a tinting solution containing 2% of standard-strength dye and 8% of *Albigen A*. The dyes used were Chlorazol Fast Pink BKS and Solophenyl Blue Green BL respectively. The tinted Celafibre was mixed in the ratio of 50 : 25 : 25 with 2½ in. staple, 3.0 filament denier bright and dull viscose rayon staple, the blend being spun to 1/18s cotton count yarn.

(2) Two batches of Celafibre-wool blend (50 : 50) were spun by conventional worsted methods, the tint being applied as follows—

The Celafibre component (5 in. staple, 4½ filament denier dull) was tinted at the gill box by means of a worsted oiling motion, the same Chlorazol Fast Pink BKS- and Solophenyl Blue Green BL-*Albigen A* liquors being used. The tinted Celafibre was then gilled and combed with an equal amount of untinted 64s wool and spun to 1/18s worsted count yarn.

The respective yarns were knitted into suitable panels, which were subjected to the following treatments—

#### 1. SWEALING TREATMENT

This test is designed to ascertain whether tinted stock is likely to become non-fugitive owing to accidental wetting by storm or otherwise.

A ½-in. test tube 6 in. long is firmly packed with the tinted yarn or loose fibre, inverted in a 100-ml. beaker containing 4 ml. of water, and left overnight. The fugitive tint should migrate by capillary action and form a strongly coloured ring about half way up the tube. It is at this point that fixation is likely to occur. The yarn or loose fibre is then removed, dried, and assessed for fixation by scouring. If fixation has occurred it is necessary to determine whether transference from Celafibre to the other component has occurred, a useful procedure being to dissolve the Celafibre in acetone, which enables a quick and easy examination to be made of the other component.

#### 2. "CONDITIONING" TREATMENT

This test is designed to ascertain whether fixation is likely to occur when the yarn on cop is allowed to acquire moisture of regain by capillary action from a damp surface.

The tinted fabrics are laid on a glass base plate, and upon each sample is placed a cylindrical stainless steel weight ¾ in. in diameter and ¾ in. long with a hole ⅜ in. in diameter in the centre. One millilitre of water is run into this hole, providing a small reservoir of water for capillary spreading along the dry fibres. The unit is left in position in a cool room for 16 hr. (overnight), and the swealed patterns are dried and scoured.

#### 3. STEAMING TREATMENT

Fixation during a so-called steaming treatment to inhibit snarling is a very well-known hazard. The conditions are not easily reproducible in the laboratory, since the live steam injected into the chamber does no more than ensure a saturated atmosphere at about 65°C.

The sample is then scoured and examined.

## EASE OF REMOVAL OF TINT

This may be assessed by means of the following treatments—

(1) **COLD-WATER WASH**—The samples are cascaded with cold water from a running tap for 2 min. If the tint is completely removed by this simple treatment, it is indeed fugitive.

(2) **SCOUR FOR CELAFIBRE-VISCOSE RAYON BLENDS**—The samples are treated for 15 min. at 70°C. in a scour bath containing 2 g. soap plus 0.3 g. soda ash per litre.

(3) **SCOUR FOR CELAFIBRE-WOOL BLENDS**—The samples are treated for 15 min. at 50°C. in 2 g. soap plus 0.3 g. soda ash per litre.

Observations on these pilot trials, together with relevant controls, are given in Tables I and II.

**Confirmatory Trials on Celafibre-Wool Blend**

Two 500-lb. batches of Celafibre (4½ filament denier, 5 in. staple) were tinted in sliver form at the gill box by the conventional worsted oiling motion, with the Albigen A complexes of the following direct cotton dyes—

Chlorazol Fast Pink BKS (ICI)  
Solophenyl Blue Green BL (Gy)

in the ratio of 4 parts of Albigen A to 1 part of standard-strength dye. One sliver end in six was run over the oiling motion, the uptake of tinting solution being 2½% on the total weight of Celafibre. The concentrations of dye and Albigen A were

again 2% and 8% respectively. The Celafibre was gilled, combed, and finisher-gilled into top form for despatch to various spinners.

Selected firms were asked to produce a 1/36s worsted count 50:50 blend from this tinted Celafibre and untinted 64s worsted wool top, the yarn to be delivered on cheese and cop—

**SPINNER No. 1**—No known fixation hazard, the yarn being spun and doubled into 2/36s worsted count on cheese for weaving trials.

**SPINNER No. 2**—This firm covered the well known cellar conditioning treatment, in which the cops from the mule were placed on a damp red-brick cellar floor so that they would take up water of regain before being passed forward to the knitter or weaver.

**SPINNER No. 3**—This firm covered the well known steaming treatment referred to previously, in which the yarn in cop form is steamed to remove "snarl" to facilitate subsequent textile operations.

The six samples were examined for fugitivity as detailed in Table III.

It will thus be seen that no fixation has occurred on either component during the commercial preparation of yarns under conditions which cover well known fixation hazards.

**Method of Application****COTTON SPINNING SYSTEM**

In spinning by the cotton system, tinting is usually carried out by means of an atomised spray.

TABLE I  
Celafibre-Viscose Staple Blend

Dye	(F ... Fugitive)	N ... Non-fugitive)	Shade achieved	Cold-water wash	Scoured after—	
					Water Swealing	Steaming
Chlorazol Fast Pink BKS (control)	...	...	Bluish red	N*	N	N
Chlorazol Fast Pink BKS-Albigen A complex	...	...	Brighter	F	F	F
Solophenyl Blue Green BL (control)	...	...	Greenish blue	N*	N	N
Solophenyl Blue Green BL-Albigen A complex	...	...	Brighter	F	F	F
Kiton Red G (acid dye for control)	...	...	Yellowish red	N*	N	N

\* Fugitive to scouring

TABLE II  
Celafibre-Wool Blend

Dye	(F ... Fugitive)	N ... Non-fugitive)	P ... Practically fugitive)	Shade achieved	Cold-water wash	Scoured after—	
						Water Swealing	Steaming
Chlorazol Fast Pink BKS (control)	...	...	...	Bluish red	N*	N	N
Chlorazol Fast Pink BKS-Albigen A complex	...	...	...	Brighter	P	F	F
Solophenyl Blue Green BL (control)	...	...	...	Greenish blue	N*	N	N
Solophenyl Blue Green BL-Albigen A complex	...	...	...	Brighter	P	F	F
Brilliant Blue 51185 (LBH) (acid dye for control)	...	...	...	Greenish blue	N*	N	N

\* Fugitive to scouring

TABLE III  
(F ... Fugitive)

Processing conditions				Spinner No. 1 From mule only		Spinner No. 2 Cellar-conditioned on cop		Spinner No. 3 Steamed on cop	
				Pink	Green	Pink	Green	Pink	Green
Tint	...	...	...	F	F	F	F	F	F
Cold-water rinse	...	...	...	F	F	F	F	F	F
Scoured at 50°C.	...	...	...	F	F	F	F	F	F

The ideal position for this is at the last hopper feed in the opening line, as at this point the fibre is in a partly open state, which permits a better distribution of the tint. An application of moisture at an earlier stage tends to consolidate the unopened fibre, and for this reason it should be avoided.

The atomised-spray principle is incorporated in various makes of apparatus, and many mills already use this method for fibre lubrication. The spray unit produced by the Manchester Oil Refinery meets all the essential requirements for this application of tint. It can be fitted in any convenient position, within a reasonable distance of the hopper feeder or other suitable hopper in the opening range. It consists of a small tank, an electric motor driving a gear pump, a pressure control valve and gauge, and an electric control. One spray unit can supply only one opening range, but several units can be fed from one main feed tank. This tank should be placed conveniently near the opening lines, giving a 9 ft. head. Additional feed tanks, one for each colour, will be needed if a variety of blends are being processed.

The spray nozzle, incorporating a small filter, is fitted inside the hopper, and directed so as to impinge on the bulk of the fibres. This nozzle is under direct pressure, supplied from the spray unit by means of small-bore tubing to the side of the hopper.

From the point of view of tint uniformity and ease of processing, the additional moisture should represent 2–2.5% the concentration of the dye solution or emulsion and the number of jets in use being adjusted to obtain this figure.

The spray stops automatically when feeding ceases, which prevents the application of an excess of dye and moisture to the fibre present in the hopper during stationary periods. Where mills are finding it advantageous to spray an emulsion of oil and water on the hopper feeder, the tint can be applied from the same emulsion.

#### WORSTED SPINNING SYSTEM

In spinning by the worsted system, tinting is usually carried out at the first gill box, and this procedure may be followed with Celafibre destined for blending with wool. The tint may be applied in aqueous solution or together with wool oil, should it be desired to apply additional lubricant, by means of the conventional lubricating roller or similar device.

#### Summary and Conclusion

In this paper we have given an outline of the considerations involved in the fugitive tinting of Celafibre for admixture with other fibres, and have suggested the use of direct cotton dye–polyvinylpyrrolidone complexes for this purpose. It would be too optimistic to suppose that they will be found to be fugitive to every variant of the numerous processes to which blended yarns and fabrics may be subjected before it is convenient to remove the tint, and it should be noted that so far our observations have extended to only a limited number of hazards affecting yarns in hank and cop form.

Only by a full appreciation of the hazards to which fugitive tints may be subjected by both

textile manufacturer and dyer and finisher will a final solution be found. The benefit accruing to the textile industry would indeed be worth while, and that there are many possibilities yet to be explored may be deduced from the contents of this paper.

\* \* \*

Our thanks are due to Messrs. W. H. Spencer & Sons Ltd. (Halifax), Thomas Burnley & Sons Ltd. (Gomersal, near Leeds), and the Mossley Wool Combing & Spinning Co. Ltd. (Mossley, near Manchester) for assistance in carrying out the bulk trials, and also to the Directors of British Celanese Ltd. for permission to publish this paper.

(MS. received 25th February 1953)

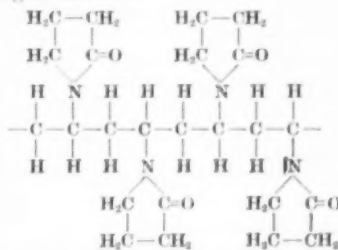
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#### Discussion

Mr. R. K. FOURNESS: Have the lecturers an explanation of this restraining action of polyvinylpyrrolidone?

Mr. OLPIN: Polyvinylpyrrolidone has the following structure—



It is thus a long-chain saturated aliphatic hydrocarbon to each alternate carbon atom of which is attached the nitrogen atom of a pyrrolidone ring. The polymer has a molecular weight range\* of 10,000–90,000, which requires an aliphatic carbon chain range of roughly 200–1600 atoms. These chains are probably considerably folded in aqueous solution.

Scale models of the polymer suggest that the pyrrolidone rings are orientated, at least in the straight-chain sections where dye fixation is most likely to occur, at right angles to the carbon chain, in such a way as to give a hydrophilic face consisting of two staggered rows of carbonyl oxygen atoms about 6 Å. apart, and spaced at about 5 Å. along the chain, the  $\text{CH}_2\text{CH}_2\text{CH}_2$  groups of the pyrrolidone rings forming a hydrophobic face on the opposite side of the carbon chain. This spacing should allow hydrogen bonding between direct cotton dye and polyvinylpyrrolidone analogous to that between direct cotton dye and cellulose at at least two points.

Mr. B. G. B. SLOCOMBE: Do the lecturers consider that tinting a blended mixture of Celafibre and wool would be as satisfactory as tinting the Celafibre portion only?

Mr. WESSON: We have restricted positive recommendations to those confirmed by bulk trials in which the Celafibre only was tinted. Laboratory trials, however, suggest that selected dyes would give satisfactory results on the blend. It will, of course, be necessary for the spinner to satisfy himself that the dyes chosen are suitable for his purpose.

Mr. R. PENDLEBURY: What is the stability of the polyvinylpyrrolidone-direct cotton dye complex? Can stock solutions be kept for any length of time?

Mr. WESSON: These combinations are very stable. Our observations suggest that certain dyes, e.g. Chrysophenine, are more soluble in polyvinylpyrrolidone solutions than in water alone. Stock solutions have shown no deterioration after two or three months' storage.

Mr. F. SMITH: At what pH was Kiton Red G applied, and would a high pH prevent fixation?

Mr. WESSON: Acid dyes used as tints for Celafibre are applied from a neutral bath. Highly alkaline solutions would introduce the risk of incipient saponification.

Mr. R. HEATON: Have any trials been done on the ease of removal of these tints from very highly twisted crêpe yarns?

Mr. WESSON: No. Our trials have not yet extended to continuous-filament yarns, but polyvinylpyrrolidone is so soluble in water that we would not anticipate any difficulties.

Mr. R. BIRCH: How do these new tints compare in light fastness with those in present use?

Mr. WESSON: The polyvinylpyrrolidone-direct cotton dye complexes will be found to have a fastness to light more than adequate for the purpose for which they are intended.

Mr. G. E. GRIFFITHS: Is polyvinylpyrrolidone likely to be held by the wool fibre, thus affecting subsequent dyeing?

Mr. WESSON: This is most unlikely. Its high molecular weight militates against its penetrating textile fibres.

Mr. J. C. THOMPSON: Has tinted cloth been subjected to crabbing and steaming as practised on worsted dress cloths? I doubt whether the steaming tests described are as severe as this.

Mr. WESSON: No. Our trials with woven goods have not yet reached the dyehouse. There is, of course, some risk of dye migration and fixation on wool and cellulose fibres during crabbing and steaming, depending upon the severity of the treatment given. Present indications are that Solophenyl Blue Green BL is better than Chlorazol Fast Pink BKS.

We can assure the questioner, however, that many of our Celafibre-wool cloths, commission-dyed in the ordinary way, have not received crabbing treatments because the dyer did not consider such treatment either necessary or desirable. Steaming as practised on worsted dress goods is more severe than the so-called "steaming" we have described, but few Celafibre-wool cloths will normally require such a drastic setting treatment.

## The Dyeing of Viscose Rayon at High Temperatures

K. BUTTERWORTH

*Meeting of the Manchester Section held at the Textile Institute, Manchester, on 20th February 1953,  
Mr. F. Farrington in the chair*

### INTRODUCTION

The use of temperatures much higher than those normally employed in dyeing has created considerable interest in recent years, and it seems opportune to review these developments, so that some assessment can be made of the potential value of these processes. This is the primary purpose of this paper, as there is little information dealing with this new aspect of dyeing published in this country, and, even if all the foreign literature is included, the published work is not extensive and, with one exception, is confined entirely to laboratory experiments.

During the last ten years dyeing methods have been developed which could in the widest sense be termed high-temperature processes, e.g. the various pad-steam and the molten-metal processes. These methods have been very thoroughly described in the literature<sup>1-3</sup> and will not be considered here. The present paper is concerned primarily with dyeing at temperatures in the region 110–120°C.

It is well established that high temperatures favour redistribution of dyes, and it is surprising that attention was not directed more seriously

toward the use of temperatures above the boil until the post-war years; yet much time is spent enabling dyes to redistribute after the initial "strike". The dyer of viscose rayon cakes, for example, regards 2–4 hr. as quite a normal dyeing time for direct cotton dyes, while 6 hr. is not unusual for those dyes which present most difficulty. Certain vat dyes, according to Burgess<sup>4</sup>, require 10½ hr. to produce an acceptable result.

Despite this incentive to find improved methods, it was 1948 before Royer and his co-workers first reported<sup>5</sup> on the effect of high temperatures on the dyeing of viscose rayon. It is well known<sup>6,7</sup> that diffusion of the dye increases with temperature, and Royer showed that this remains true above 100°C., complete fibre penetration being obtained with certain direct dyes in 50 sec. at 140°C.

A report on high-temperature dyeing in 1949<sup>8</sup> confirmed this greater rate of dyeing, illustrated the improved levelling of direct cotton dyes, and stated that of the vat dyes tested all except those of the Caledon Jade Green type are unstable when dyed at 120°C.

Both these papers<sup>5,8</sup> were based entirely upon small-scale laboratory dyeings, and it remained for Steverlynck of Groeninghe Ververij and his co-worker, Drijvers, to devise machinery for dyeing above 100°C., which gave satisfactory results on viscose rayon cakes in 1–2 hr. with both direct and vat dyes, this work being the subject of two British patents<sup>9,10</sup>.

Since the publication by Drijvers<sup>11</sup> of the work done at Groeninghe Ververij, the attention of various machinery manufacturers has been directed towards the production of machines suitable for dyeing packages above 100°C., and numerous machines are available. A novel development is the Barotor machine, announced recently by du Pont for dyeing piece goods at high temperatures<sup>12</sup>. Although it is primarily intended for the newer synthetic fibres, it will presumably be suitable also for rayon piece goods, while jig-type machines designed for the same purpose are available.

It cannot be doubted that these methods are becoming of increasing importance, and the results which follow show the effect of dyeing at temperatures in the region of 110–130°C. on direct and vat dyes, on the viscose rayon itself, and on the levelness and the fastness of the resultant dyeings. The advantages of high-temperature dyeing have been illustrated by dyeings carried out on viscose rayon cakes, since it is in this field that the greatest difficulty is encountered in regard to levelness. Nevertheless, the information presented will be of value for a consideration of the dyeing of viscose rayon at high temperature in any of its many forms.

#### DIRECT COTTON DYES

##### Levelling Effect

According to the Society's classification of direct cotton dyes<sup>13</sup> the least difficulty in producing level and well penetrated dyeings is to be expected with the self-levelling dyes of Class A, and the greatest difficulty with dyes of Classes B and C, which are salt- and temperature-controllable respectively. This is borne out by practical experience, and it is primarily for dyes in the latter classes that improved dyeing methods are required. It is interesting, therefore, to ascertain the degree of improvement in redistribution which is realised by increasing the temperature from 85° to 120°C.

Five dyes of Classes B and C were dyed on viscose rayon filament yarn in 1% depths. The dyed yarn was then knitted alongside an equal weight of undyed yarn, and these knittings were subjected to the levelling treatments indicated in Table I, the liquor to goods ratio being 50:1 in all cases. The higher temperature (120°C.) was attained by use of a suitable laboratory autoclave, the degree of levelling being assessed with the aid of the Society's Geometric Grey Scale<sup>14</sup>.

An examination of these results (Table I) shows that a considerable improvement has been obtained in nearly all cases at the higher temperature, but it is noticeable that the greatest improvement is obtained when the levelling is carried out in two stages, electrolyte being added during the latter half of the treatment.

TABLE I  
Levelling of Direct Dyes at High Temperatures  
(Contrast between originally dyed and originally undyed portions)

Treatment—										
Glauber's salt, % Na <sub>2</sub> SO <sub>4</sub>	—	—	10	10	—	10	—	10	—	10
Time, min.	...	...	30	30	30	30	30	30	30	30
Temperature, °C.	...	...	85	120	85	120	85	120	85	120
Dye	Class <sup>13</sup>									
Chlorazol Brown MS	C	2	4*	1-2	4-5	3	5			
Durazol Brown BRS	C	1-2	4*	1-2	4-5	4	5			
Durazol Blue 2GNS	B	1	1*	2	3-4	2	5			
Durazol Grey VGS	B	1	1	1	3	1	3-4			
Durazol Rubine BS	B	1	1	1-2	2	1-2	3-4			

\* Much reduced in depth

##### Dye Stability

One of the major controlling factors in dyeing at high temperature is the stability of the dyes to the particular conditions employed. The tendency of certain direct dyes to decompose when dyed at the boil has been reported by Ashpole *et al.*<sup>15</sup> and by Armfield<sup>16</sup>. This decomposition is caused by the reducing action of cellulose in the presence of alkali. Both these investigations showed that the destructive effect may be overcome by the addition of ammonium sulphate, which corrects this alkalinity. Drijvers<sup>11</sup> reported the same problem, and found oxidising agents such as perborates and hydrogen peroxide to be advantageous.

A number of direct dyes were examined by dyeing on viscose rayon filament yarn for 30 min. at temperatures varying from 85° to 130°C. in distilled water with an addition of 10% anhydrous sodium sulphate and with and without an addition of 1 g. soda ash per litre. After dyeing at the appropriate temperature, the baths were cooled to 85°C. and dyed at this temperature for 30 min. to bring them to approximately the same equilibrium. Finally the exhaustion was assessed by means of exhaust dyeings dyed for 30 min. at 85°C.

The decomposition in the control solution, i.e. in absence of fibre, was determined by means of the G.E.C. Hardy recording spectrophotometer.

Examined in this way, the dyes fall into three groups—

GROUP I—Those dyes which are stable when dyed at high temperatures under neutral conditions and which also show considerable resistance to decomposition under alkaline conditions. These stable dyes are typified by Durazol Orange 2G (Table II).

GROUP II—The second group consists of those dyes which, although not unduly affected when dyed at high temperatures in the absence of alkali, are completely destroyed in alkaline solution. Durazol Blue 2R (Table II) is representative of this group.

GROUP III—Dyes in the third group are decomposed under both neutral and alkaline conditions. It is noteworthy that dyes containing the urea linkage are prominent in this group, but not all dyes containing urea groupings are decomposed. Although only a small group, the dyes in it exhibit several minor differences. Chlorazol Fast

TABLE II  
Effect of Temperature and Alkali on Colour and Exhaustion of Direct Dyes  
(Addition of 1 g. soda ash per litre)

Temp. (°C.)	Without Addition Colour	Exhaustion	With Addition Colour	Exhaustion
(I) 2% DURAZOL ORANGE 2GS				
85	Control	Control	Little stronger, trace redder	Trace more
100	Trace weaker	Trace less	Little stronger, little redder	Trace more
110	Little weaker	Little less	Little stronger, little redder	Trace more
120	Little weaker	Little less	Little stronger, little redder	Trace more
130	Little weaker	Little less	Little stronger, redder, duller	Trace more
(II) 2% DURAZOL BLUE 2RS				
85	Control	Control	Equal	Equal
100	Little stronger, little redder	Little more	Much weaker, much redder, much duller	Dye decomposed. Exhaustion not assessed.
110	Equal	Little more	Pale violet	
120	Equal	Little more	Pink	
130	Little stronger, little redder	Little more	—	
(III) 2% CHLORAZOL FAST PINK BKS				
85	Control	Control	Equal	Equal
100	Trace weaker	Trace less	Trace weaker	Equal
110	Little weaker	Equal	Weaker	Little more
120	Weaker	Little more	Much weaker	More
130	Much weaker	Much more	Very much weaker	Much more

Pink BK may, however, be taken as an example (Table II).

The decomposition of direct dyes in the absence of cellulose at high temperature does not necessarily exactly follow the above classification, though there is a general correlation. Durazol Orange 2G is unaffected under both neutral and alkaline conditions, while the results for Durazol Blue 2R and Chlorazol Fast Pink BK are given in Fig. 1.

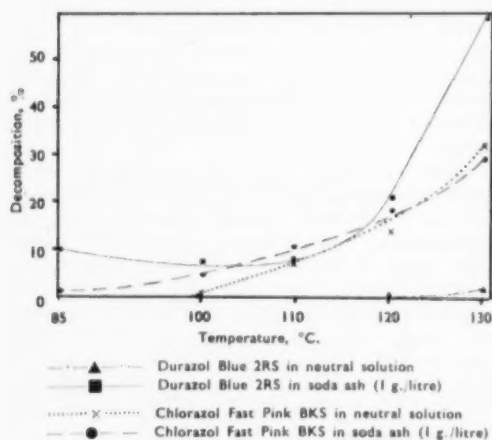


FIG. 1.—Decomposition of Direct Dyes in Absence of Cellulose

In Table III a selection of direct dyes has been grouped according to the above classification. The pH of solutions of the dyes in distilled water at average dyebath concentration are also given.

TABLE III  
Stability of Direct Dyes to High-temperature Dyeing

GROUP I	pH
Durazol Yellow G ... ..	6.5
Durazol Yellow 4G ... ..	8.2
Durazol Yellow 6G ... ..	7.6
Durazol Yellow GR ... ..	6.8
Durazol Yellow 3R ... ..	6.4
Durazol Orange 2G ... ..	7.0
Chlorazol Orange PO ... ..	7.3
Chlorazol Orange Brown X ... ..	7.0
Benzopurpurine 4B ... ..	7.0
Benzopurpurine 10B ... ..	7.8
Chlorazol Brilliant Purpurine 8B ... ..	7.2
Durazol Blue 8G ... ..	6.9
GROUP II	
Chlorazol Diazo Yellow 2G ... ..	6.6
Chlorazol Yellow G ... ..	6.6
Chlorazol Yellow 6G* ... ..	6.7
Chrysophenine G* ... ..	7.1
Durazol Flavine R ... ..	6.3
Chlorazol Fast Orange G ... ..	4.0
Chlorazol Brilliant Orange 3R ... ..	7.9
Chlorazol Diazo Orange 3G ... ..	6.3
Chlorazol Diazo Orange 2G ... ..	8.2
Durazol Orange R ... ..	6.8
Durazol Orange 4R ... ..	7.1
Icyl Orange G ... ..	6.8
Chlorazol Brown M ... ..	7.6
Chlorazol Brown B ... ..	6.6
Chlorazol Brown GM ... ..	6.9
Chlorazol Brown LF ... ..	7.1
Chlorazol Brown PB ... ..	6.8
Chlorazol Brown PRK ... ..	7.2
Chlorazol Orange Brown RN ... ..	6.6

TABLE III—continued

	pH
Chlorazol Orange Brown 2R	6.4
Chlorazol Catechine BN	9.4
Chlorazol Catechine GR	8.4
Chlorazol Drab RH	7.0
Durazol Brown G	6.7
Durazol Brown BR	7.6
Durazol Brown 8R	6.4
Durazol Orange Brown G	7.0
Durazol Orange Brown 2R	5.2
Durazol Red Brown 4RN	6.8
Icyl Brown G	6.8
Chlorazol Fast Red F*	7.2
Chlorazol Diazo Scarlet R	6.7
Chlorazol Diazo Scarlet 2R	7.6
Chlorazol Rose B	6.6
Chlorazol Bordeaux B	6.6
Chlorazol Bordeaux 6B	6.7
Chlorazol Diazo Bordeaux 2B	7.3
Chlorazol Corinth GW	8.4
Durazol Scarlet 2G	6.7
Durazol Rubine B	6.9
Chlorazol Fast Helio 2RKK	6.1
Chlorazol Violet WB	6.7
Chlorazol Violet N	7.2
Chlorazol Violet R	6.7
Durazol Helio B	7.3
Durazol Violet 2B	7.1
Chlorazol Blue G	5.8
Chlorazol Blue B	6.8
Chlorazol Blue 2R*	7.1
Chlorazol Blue RW	7.7
Chlorazol Sky Blue FF	6.7
Chlorazol Sky Blue GW	6.6
Chlorazol Azurine G	8.4
Chlorazol Steel Blue 6B	7.8
Chlorazol Copper Blue B	7.6
Chlorazol Copper Blue 2B	7.9
Chlorazol Copper Blue FB*	7.9
Chlorazol Dark Blue B	7.4
Chlorazol Diazo Blue BR	7.7
Chlorazol Diazo Blue 2R	7.2
Durazol Blue G	7.6
Durazol Blue 2GN	7.2
Durazol Blue 4G	6.0
Durazol Blue 2R	7.6
Durazol Blue 3R	7.0
Durazol Blue 4R	6.9
Durazol Blue 8R	7.6
Icyl Blue G	6.8
Icyl Sky Blue G	7.6
Icyl Blue 2R	6.7
Chlorazol Green G	8.0
Chlorazol Green BN	7.3
Chlorazol Dark Green PL	7.4
Chlorazol Diazo Green BD	6.3
Durazol Grey B	7.3
Durazol Grey VG	8.4
Durazol Grey N	7.7
Chlorazol Viscose Black B	9.1
Chlorazol Black E	7.7
Chlorazol Black EN	7.5
Chlorazol Black GF	8.0
Chlorazol Black JH	7.2
Chlorazol Black LF	7.5
Chlorazol Black PB	7.3
Chlorazol Black ZV	7.4
Chlorazol Diazo Black D	7.2

TABLE III—continued

	pH
Chlorazol Diazo Black SD	8.2
Chlorazol Diazo Black 3B	6.7
Icyl Blue Black 6B	6.7
GROUP III	
Chlorazol Fast Orange R	6.8
Icyl Orange R	6.9
Chlorazol Fast Scarlet 4BA	6.6
Chlorazol Fast Scarlet 4B	6.6
Chlorazol Fast Scarlet 8B	7.1
Chlorazol Fast Scarlet G	6.9
Chlorazol Fast Pink BK	7.8
Durazol Brilliant Red B	5.7
Durazol Red 2B	6.4
Durazol Red 6B	6.2
Chlorazol Diazo Blue 3G	7.2
Durazol Grey RG	8.1
Chlorazol Black BH	7.0

\* These products possess some resistance to alkaline conditions, but not sufficient to place them in Group I.

An arbitrary classification of this nature always creates problems relating to the allocation of borderline dyes. For example, Durazol Grey RG has been placed in Group III, as it is slightly decomposed under neutral conditions. It is, however, known that this product is being used for high-temperature work. Similarly, there are some dyes in Group II (marked with an asterisk (\*)) which possess some degree of resistance to decomposition under alkaline conditions.

The pH of the dyebaths in a few cases is distinctly alkaline, and yet some of these dyes are placed in Group II. Clearly, in these cases the amount of alkali introduced by the dye itself is not sufficient to cause decomposition, but, as pointed out by Armfield<sup>16</sup>, alkali introduced by one dye may well decompose another when the two products are used in admixture. Consequently it is advisable to take precautions when compelled to use dyes which are known to be a source of alkalinity.

It must be emphasised that this classification should be used only as a guide. Depth of dyeing, quality of the rayon used, efficiency of preparatory treatments, and time and temperature of dyeing are a few of the other factors which influence the stability of the dye.

It is noteworthy that, in contradistinction to leuco vat dyes, cellulose exerts no protective effect on the decomposition of direct dyes.

When the shade of the cake dyeings, carried out at 120°C., details of which are given later (p. 366), was compared with that of hank dyeings carried out at 85°C., a somewhat greater variation was noted than was indicated by the classification described above. This is not altogether surprising, as the conditions were quite different, but it may be that some shade variation will have to be tolerated. This should not be a serious disadvantage, providing that the variation is not too great and that it is not accompanied by a reduction in fastness properties.

TABLE IV  
Effect of Protective Agents

(2% Durazol Blue GS + 1 g. soda ash per litre)		
Protective Agent (per litre)	Temp. (°C.)	Shade
—	85	Control
—	100	Very much weaker
3 c.c. H <sub>2</sub> O <sub>2</sub> (10 vol.)	85	Equal
	100	Much weaker
	110	Very much weaker
3 g. Na percarbonate	85	Equal
	100	Trace weaker, little redder
	110	Duller, redder, weaker
3 g. Na perborate	85	Equal
	100	Trace stronger
	110	Weaker, duller, redder
1 g. (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	85	Trace stronger
	100	Trace stronger
	110	Little stronger, brighter, little redder
3 g. K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	85	Trace stronger, little redder
	100	Little redder
	110	Little redder

#### Protective Agents

As stated earlier, the decomposition due to alkaline reduction may be avoided by means of either pH control or the judicious addition of oxidising agents. The protection which may be afforded in this manner can be simply demonstrated by creating conditions which ensure decomposition and then examining the effect of appropriate additions to the dye bath.

Durazol Blue GS is a suitable dye for this purpose, and the protective effect of various agents (Table IV) was demonstrated by dyeing viscose filament yarn for 30 min. in 2% depths (liquor to goods ratio 50 : 1) at 85°, 100°, and 110°C. with addition to the dye bath of 1 g. soda ash per litre and 10% anhydrous sodium sulphate, with and without the protective agent.

It must be emphasised that this protective effect is confined to the decomposition caused by alkaline reduction, and is of no value in preventing the thermal decomposition exhibited by dyes in Group III.

A further aspect which must be continually borne in mind is that direct dyes in general are not very resistant to oxidising agents, and there is always a danger that excessive additions of these compounds will have an adverse effect on the shade. The possible extent of such damage is illustrated in Table V. The four dyes were dyed in 1% depths for 30 min. at 85°C. with an addition of 10% anhydrous sodium sulphate, a liquor ratio of 50 : 1 being employed, and the effect on the shade of the addition to the dye bath of the oxidising agents listed above was determined.

In addition, it must be remembered that both sodium percarbonate and sodium perborate yield alkaline solutions, and once the available oxygen has been used the increased alkalinity may result in still greater decomposition. Metallisable direct dyes may also chelate with potassium dichromate. The effect of these agents on the yarn strength is discussed in the final section of the paper.

Ammonium sulphate is to be preferred as a protective agent to an oxidising agent, though the best solution is without doubt to avoid alkaline conditions altogether. When the use of ammonium sulphate cannot be avoided, account must be taken of its influence as an electrolyte on the absorption of the dye.

#### Viscose Cake Dyeings

The use of high-temperature techniques is likely to be of most value in package dyeing, particularly for dyeing viscose rayon cakes. The problems associated with this system of dyeing are due in the main to the difficulty of redistributing the dye which is initially absorbed on the more accessible portions of the cake. It has been shown in Table I that the redistribution of direct dyes is improved to a considerable extent at elevated temperatures, particularly if the use of high temperatures is accompanied by salt control.

Viscose rayon cake dyeings obtained with four dyes have been chosen for comparison. These four dyes have been selected as varying widely both in general dyeing properties as indicated by the Society's classification<sup>13</sup> and in the particular field of cake dyeing as indicated by the classification given by Douglas<sup>17</sup>. This latter classification groups direct dyes into four groups numbered 1–4, where 1 indicates maximum and 4 minimum suitability. The dyes and their classification according to the above systems are given in Table VI.

Experience at normal temperatures is that medium depths of direct dyes require from 2 hr. for Group 1 up to 6 hr. for Group 4 dyes in order to produce well penetrated cakes. Indeed, certain dyes in Group 4 cannot be applied satisfactorily even with these extended dyeing times, and certainly not in pale depths. It is obvious that it is with those dyes presenting greatest difficulty when applied by existing methods that there lies most need for improvement. In addition, such methods of dyeing will also enable increased production to be realised with all classes of dyes.

These dyeings were carried out on viscose rayon cakes (150 denier, 27 fils) in a laboratory pressure circulating machine in which the high temperatures are made possible by the introduction of a static

TABLE V  
Effect of Oxidising Agents on Direct Dyes

Oxidising Agent (per litre)	1% Chlorazol Brown MS	1% Chlorazol Fast Red FS	1% Durazol Orange 2GS	1% Durazol Blue 2GNS
—	Control	Control	Control	Control
3 g. Na percarbonate	...	Much weaker	Weaker	Very much weaker
3 g. Na perborate	...	Much weaker	Weaker, greener	Very much weaker
3 c.c. H <sub>2</sub> O <sub>2</sub> (10 vol.)	...	Little weaker	Little weaker, little redder	Weaker
3 g. K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	...	Weaker	Little weaker	Much weaker

pressure above 1 atmosphere into the dyeing chamber. A liquor ratio of 8:1 was employed, and the direction of flow was reversed every 5 min.

Chlorazol Brown MS was dyed in a 1% depth in this machine for 40 min. without addition of electrolyte at 90° and 120°C. The result obtained at the latter temperature was a considerable improvement over those obtained at 90°C. By increasing the dyeing time to 1 hr. a level dyeing was obtained at 120°C., but exhaustion was only 70%. Addition of 4% anhydrous sodium sulphate at the commencement of dyeing gave a very unlevel result, though this was still slightly superior to the 40 min. dyeing at 90°C. without electrolyte. By adding the electrolyte over the last 30 min. a level dyeing was obtained. This procedure was preferred to the alternative method of cooling the bath and allowing the dye to feed on slowly, as it utilises the greater redistributive powers obtaining at the higher temperature.

It is not always possible to use this procedure, since in some cases excessive quantities of salt are required to obtain an economic exhaustion at 120°C. For example, 1% Durazol Flavine RS dyed at 120°C. required 20% salt to give 80% exhaustion with an 8:1 liquor ratio, and in such cases a combination of electrolyte and temperature control is to be advised, otherwise precipitation of the dye may be encountered.

An identical series of experiments with Durazol Brown BRS gave similar results.

Durazol Rubine BS gave a level result when dyed for 1 hr. at 120°C., exhaustion being controlled by the gradual addition of 8% anhydrous sodium sulphate over the final 30 min. This result may seem to be at variance with the results obtained in the redistribution tests given in Table I, which show that this dye does not redistribute well even at 120°C. Durazol Rubine B, however, has a very low exhaustion in the absence of electrolyte, and, in this case particularly, levelness depends in large measure on careful salt control.

A dyeing of 1% Chlorazol Orange POS at 120°C. for 1 hr., 4% anhydrous sodium sulphate being added over the last 30 min., was unlevel. This result, which has been confirmed by repeat tests, is difficult to understand. While it is true that this dye has a rapid rate of dyeing at 90°C., and its rate of migration is not high for Class A<sup>13</sup>, nevertheless levelling tests at 120°C., at admittedly a longer liquor to goods ratio (30:1) but with equal electrolyte concentration in the bath, indicate that 30 min. at this temperature should be sufficient for a level cake to be obtained. It has not been possible to investigate this unexpected behaviour fully, as attention has been directed, for the reasons already stated, towards producing level results with those dyes which present most problems. It is interesting to note, however, that Durazol Flavine RS, also a Class A dye, showed a similar behaviour.

It is well established practice to add levelling agents to the dyebath when dyeing direct dyes, and a dyeing of Chlorazol Orange POS at 120°C. for 1 hr. with 5% Dispersol VL, 4% anhydrous

sodium sulphate being added over the last 30 min., was perfectly level.

The results of the cake dyeings which have been described are summarised in Table VI.

TABLE VI  
High-temperature Dyeing of Direct Dyes on  
Viscose Rayon Cakes (150/27)

Temp. (°C.)	Time (min.)	Na <sub>2</sub> SO <sub>4</sub> Strength (%)	Time added (min.)	Levelness
1% CHLORAZOL BROWN MS (C, 4)*				
90	40	0	—	Very unlevel
120	40	0	—	Sl. unlevel
120	60	0	—	Level
120	60	4	0	Very unlevel
120	60	4	30-60	Level
1% DURAZOL BROWN BRS (C, 3)				
90	40	0	—	Unlevel
120	40	0	—	Sl. unlevel
120	60	0	—	Level
120	60	4	0	Very unlevel
120	60	4	30-60	Level
1% DURAZOL RUBINE BS (B, 2)				
120	60	0	—	Level (very poor exhaustion)
120	60	8	30-60	Level
1% CHLORAZOL ORANGE POS (A, 1)				
120	60	4	30-60	Unlevel
120	60	4†	30-60	Level

\* Letters in parentheses indicate S.D.C. classification<sup>12</sup>, and figures cake-dyeing class<sup>13</sup>.

† Dyebath contains also 5% Dispersol VL.

The light and wet fastness properties of a selection of direct dyes dyed at high temperature are given in Table VII compared with those of dyeings at 85°C.

The experience which has been gained suggests that, if account is taken of the greater difficulties involved in dyeing pale depths and dye mixtures, dyeing times up to 1½ hr. will be required for dyeing viscose rayon cakes with direct dyes at 120°C. Non-ionic levelling agents of the Dispersol VL type have been found to be very valuable under these conditions.

Summarising the results obtained with direct dyes, it may be stated that high-temperature dyeing permits equal or better results to be obtained in much shorter times than are normally employed. The use of high-temperature processes, however, is not free from problems, and will demand a greater degree of control by the dyer. Clearly, since direct dyes in general are so easily destroyed by the reducing action of the alkali-cellulose system, it is essential to exclude wherever possible all sources of alkalinity. Apart from the water supply, alkali may be introduced by the dye, the fibre, and dyebath assistants. It is advisable as a final precaution to check the dyebath pH before commencing dyeing, so that ammonium sulphate may be added if necessary. Checking of the pH during the dyeing will also be necessary in cases where softened water is used.

#### VAT DYES

It will be realised that the problems of dyeing vat dyes at high temperatures are considerable.

TABLE VII  
Effect of High-temperature of Dyeing on Light and Washing Fastness of Direct Dyes

Dye	Temp. of Dyeing (°C.)	Washing Fastness				Light Fastness
		Repeated Wash at 40°C.	Staining of Viscose Rayon	Repeated Wash at 60°C.	Staining of Viscose Rayon	
Chlorazol Orange POS...	85	3-4	3	2	2	4-5
	120	Little weaker	3	Much weaker	2	4-5
		3-4		Very much weaker		
Durazol Rubine BS ...	85	4	4-5	3	4-5	6-7
	120	4-5	5	Weaker	4-5	6-7
				Little weaker		
Durazol Brown BRS ...	85	3-4	3	2	2	6
	120	Trace weaker, trace yellower	3	Much weaker	2	6
		4				
Chlorazol Brown MS ...	85	3-4	3	2	2	5
	120	Trace weaker, trace bluer	3	Much weaker	2	5
		4				
				Much weaker		

The general instability of alkaline leuco compounds when dyed even at 80-90°C. is all too well known, and various attempts have been made to prevent this decomposition by the use of e.g. dextrins<sup>18</sup>, inorganic nitrites<sup>19</sup>, hydroxylamine and similar products<sup>19</sup>, and inorganic chlorates<sup>19</sup>.

It is generally considered that the viscose rayon itself is adversely affected when treated above 80°C. in caustic soda solutions of dyebath strength. Hampson<sup>20</sup> reports a drop of approx. 30% in the wet strength of 150/27 viscose filament yarn when dyed with vat dyes for 2 hr. at 85°C. as compared with 80°C. On the other hand, the 1949 report<sup>8</sup> states that, although the vat dyes in general were decomposed when dyed at 120°C., the viscose rayon was unaffected.

In the face of these conflicting statements, it seemed worth while examining the effect of high temperature on the alkaline leuco process. Eleven vat dyes were selected which possess appreciable resistance to over-reduction. They were dyed for 30 min. at 120°C. and then for 30 min. at the normal dyeing temperature. The dyeings obtained were compared for colour with dyeings dyed for 60 min. in the usual manner. The results for the eleven dyes (Table VIII) show that seven are substantially unaffected—

Caledon Yellow GN  
Caledon Golden Yellow GK  
Caledon Gold Orange GN  
Caledon Red BN  
Caledon Violet XBN  
Caledon Jade Green XBN  
Caledon Dark Blue G

A number of experiments have been carried out on viscose rayon cakes, and the much improved levelling obtained at 120°C. has been demonstrated. The higher temperature alone, however, is not sufficient to yield a perfect result, and the use of a levelling agent such as Dispersol VL is still necessary. Thus, for example, 0.5% FD Caledon Jade

TABLE VIII  
Effect of High-temperature Dyeing on Colour of Vat Dyeings

Caledon Dye	Dyeing Temp. (°C.)	Colour
Yellow GN ...	50	Control
	120	Little stronger, little redder
Golden Yellow GK	50	Control
	120	Trace weaker
Gold Orange 3G ...	50	Control
	120	Much redder
Gold Orange GN ...	50	Control
	120	Little weaker
Brown R ...	50	Control
	120	Duller, little weaker
Red BN ...	20	Control
	120	Little stronger
Red Violet 2RN	20	Control
	120	Weaker
Violet XBN ...	50	Control
	120	Little bluer
Jade Green XBN...	50	Control
	120	Equal
Olive Green B ...	50	Control
	120	Yellower
Dark Blue G ...	50	Control
	120	Trace weaker

Green XBN Powder Fine gives a grossly unlevel result when dyed on viscose rayon cakes at 50° or 80°C. for 30 min., but at 120°C. a very great improvement was effected, and at 120°C. with an addition of 5% Dispersol VL a nearly level dyeing was obtained. The effects of high-temperature dyeing on the light and washing fastness of FD Caledon Jade Green XBN Powder Fine and Caledon Red BN 200 Paste Fine were determined, and no appreciable differences were noted.

While these results are of considerable interest, it is doubtful whether a wide range of stable vat dyes will be found from available products. Attention must naturally be directed towards the use of higher temperatures in the pigmentation of

TABLE IX  
Effect of High-temperature Vat Dyeing on Tensile Strength of Viscose Rayon (150/27) Yarn

Caledon Dye	Dyeing Temp. (°C.)	Dry Tests		Wet Tests	
		Tenacity (g./denier)	Extension (%)	Tenacity (g./denier)	Extension (%)
Untreated ...	—	2.1	18	1.0	26
Water ...	85	2.0	20	1.0	26
	120	1.9	22	1.0	24
Violet XBN ...	50	2.1	20	1.2	19
	120	1.9	21	0.9	18
Jade Green XBN ...	50	2.0	21	1.0	22
	80	2.0	20	1.0	22
	120	2.0	20	1.0	20
Red BN ...	20	2.0	21	1.1	21
	120	1.7	18	0.8	18

TABLE X  
Effect of Protective Agents on Tensile Strength of Viscose Filament Yarn at High Temperatures

Protective Agent (per litre)	Temp. (°C.)	Dry Tests		Wet Tests	
		Tenacity (g./denier)	Extension (%)	Tenacity (g./denier)	Extension (%)
Untreated ...	—	2.1	23	1.1	26
Water ...	85	2.2	20	1.1	22
	120	2.2	22	1.1	20
3 g. Na percarbonate	85	2.1	20	1.0	20
	120	1.8	20	0.8	17
3 g. Na perborate ...	85	2.2	20	1.0	20
	120	1.9	21	0.9	18
3 c.c. H <sub>2</sub> O <sub>2</sub> (10 vol.)	85	2.1	21	1.1	23
	120	2.0	21	0.8	16
3 g. K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ...	85	2.2	20	1.0	24
	120	2.2	22	1.1	22
1 g. (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ...	85	2.2	19	1.0	22
	120	2.2	22	1.1	24

viscose rayon cakes when dyed with vat dyes by the Abbot-Cox process, since savings in time may possibly be effected by this means.

Work along these lines is proceeding, and preliminary results are promising and indicate that finely dispersed powder brands, e.g. the FD Caledon Powders Fine, are to be preferred to the Paste Fine qualities. Adequate control appears to be possible with the FD brand without dispersing agent, pigmentation being complete in 1 hr. Dispersol VL has an aggregating effect on the FD brands above 100°C. The Paste Fine brands, however, are easily aggregated above 100°C. irrespective of whether Dispersol VL is used or not.

#### EFFECT OF HIGH TEMPERATURE ON THE TENSILE STRENGTH OF VISCOSE RAYON

The wet and the dry tensile strengths of viscose filament yarn treated for 30 min. at 85° and 120°C. in distilled water and also dyed for 30 min. with vat dyes at normal temperatures and at 120°C. have been determined. The impression from these results (Table IX) is that yarn dyed up to 120°C. with direct dyes should suffer no serious loss in strength. With vat dyes dyed by the alkaline leuco process, however, the yarn may lose 10–20% in dry and in wet tensile strength.

The results obtained are not considered to be sufficiently extensive to condemn the leuco process out of hand, and a final opinion must be left until it is possible to carry out a statistical survey.

The effect of the protective agents for direct dyes on the tensile strength has been determined,

by treatment for 30 min. at 85° and 120°C. in distilled water and in solutions of the agents. The results (Table X) serve to emphasise the preference for pH control already expressed.

#### CONCLUSION

It will be apparent from all that has been said that much work remains to be done. The advantages of high temperatures for dyeing in package form are now well established. The possibilities in piece dyeing have not been seriously examined, and it is certain that in this direction lies a field for major exploration.

\* \* \*

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### Discussion

Mr. F. FARRINGTON: Can the alkaline reduction of direct dyes be prevented by the use of Resist Salt L (nitrobenzenesulphonic acid)?

Mr. BUTTERWORTH: Resist Salt L has been examined for this purpose, and found to be of little value.

Mr. A. H. NORRIS: How does ammonium sulphate act as a dyebath assistant to remove alkalinity, since ammonia cannot leave the enclosed high-pressure system?

Mr. BUTTERWORTH: It is difficult to determine how ammonium sulphate functions. Measurements of pH before and after dyeing are not of much assistance, since the important factor is the pH at the high temperature.

Dr. C. M. WHITTAKER: Would it not have been better when testing direct cotton dyes to have used the more concentrated brands which the intelligent cake dyer uses, so as to have greater control of the additions of electrolyte?

Mr. BUTTERWORTH: The use of S brands was deliberate, as it was considered that if these could be applied satisfactorily much less difficulty would be encountered with the concentrated brands, which are, of course, especially suitable for cake dyeing.

Dr. WHITTAKER: The fact that there is no such thing as a standard viscose rayon cake makes comparative studies of dyes very difficult. Before a cake is used, it is recommended that small

lengths of yarn from the extreme outside and inside of the cake should be wound off and tested for dyeing variation under normal dyeing conditions. If markedly different dyeing affinity is shown, then that cake should be rejected, or otherwise a wrong conclusion might be drawn about the dye under test.

Mr. BUTTERWORTH: The physical and chemical variations experienced with viscose rayon cakes are well known to dyers of these packages. During the present investigation much improved levelness has invariably been obtained at the high temperatures, and indeed the results could not have been obtained at normal temperatures, whatever precautions had been taken as regards cake selection.

Mr. S. BURGESS: Large quantities of viscose rayon yarn in cake form are dyed with Durazol Grey RG and Durazol Red 2B, which appear in the lowest group of dye stability. In conjunction with Chlorazol Orange PO and similar dyes, the dyes named are very widely used, and the admission of their instability should be a spur to dye manufacturers to replace them with dyes which are superior as regards resistance to reduction in the dyebath.

Chlorazol Orange PO has been indicated as behaving rather out of character for a Class A dye, and sometimes giving unexpectedly unlevel results. This confirms bulk experience, and it is necessary to treat Chlorazol Orange PO as a Class B dye and use very careful salting technique. It should be remembered that Class A dyes are so under the specified conditions of the test. Pale shades of Class A dyes will not necessarily level well when dyed on viscose rayon packages.

Mr. BUTTERWORTH: There is no disagreement with the remarks about Chlorazol Orange PO.

The reason for the use of the Class A dyes mentioned is, economic considerations apart, their level dyeing characteristics. The improved leveling obtained with Class B and Class C dyes in the region of 120° C. makes it possible to obtain level results with many of these dyes in 1–1½ hr. even in pale shades, so that in fact the dyes for which Mr. Burgess asks are available.

## COMMUNICATION

## Further Observations on the Effects of Evaporating Water from Cotton Cellulose

G. W. MADARAS and H. A. TURNER

The experiments described in a previous paper<sup>1</sup> have been extended with the object of excluding gaseous oxygen more rigorously from the system. When the purified cotton cloth was first maintained for three days in a high vacuum, and then brought into contact with chemically deoxygenated water in an atmosphere of purified nitrogen, evaporation produced a brown line, closely resembling that already described, at the boundary between wet and dry cloth. With a modified procedure, when evaporation took place *in vacuo*, no brown coloration was observed, but evidence was obtained that chemical modification of cellulose in the boundary region was still taking place.

## Introduction

The tendering which occurs when cellulosic materials are exposed to the joint action of air, light, and moisture is generally ascribed to oxidation. The mechanisms which lead to the formation of oxycelluloses have been shown, upon close examination, to be complicated, and the properties of the reaction products to be dependent upon the conditions under which the oxidation has taken place<sup>2-4</sup>. Evidence pointing to an oxidative modification of cellulose when water evaporates from it was first given by Bone<sup>5</sup>, and more recently a fuller account of this investigation was given by Bone and Turner<sup>1</sup>. In particular, they described the conditions under which the distinctive "brown line" is formed in a sharply restricted boundary region, between wet and dry cloth, when a strip of purified cotton cloth is suspended vertically with the lower end in water. In this system, the steady rise of water up the cloth is ultimately balanced by evaporation from the wetted surface.

The occurrence of brown products in the boundary region may come from reactions essentially confined to this region, and requiring cellulose, water, and atmospheric oxygen to be simultaneously present. It may, however, be due to reactions taking place over the whole of the wetted area of the cloth, the products of the reactions being transported to the boundary by ascending currents of water. Since the original work was done, W. D. Appel *et al.*<sup>6</sup> have published an account of experimental studies in the same field. In this, the principal findings of Bone and Turner are substantially confirmed, and evidence is brought forward to show that the action by which cellulose is modified is confined to the narrow boundary region.

In the experiments of Bone and Turner, the formation of a brown line may, therefore, have been due to local oxidation of the cellulose, especially as no rigorous attempts were made to exclude oxygen from the system. Additional support for this suggestion is given by the observation that a non-transportable modification of the cellulose, with enhanced reducing and acidic properties, is formed at every level on the strip of cloth at which a boundary between wet and dry has existed for any length of time.

To find out how far the presence of atmospheric oxygen is responsible for the formation of the brown line and related phenomena, the evaporation has been repeated in a closed system, in an

atmosphere of nitrogen, efforts being made to remove oxygen as completely as possible from the nitrogen, the cellulose, and the water. It was thought that a brown line might not then be formed, or that, under specified conditions, the intensity of the colour might at least be lessened. These hopes were not fulfilled, and the results obtained were inconclusive. However, by conducting the evaporation *in vacuo*, i.e. by avoiding the introduction of nitrogen, even when highly purified, it was possible to inhibit the formation of the brown coloration. Nevertheless, other evidence of local modification of the cellulose, such as enhanced methylene blue absorption, was still obtained.

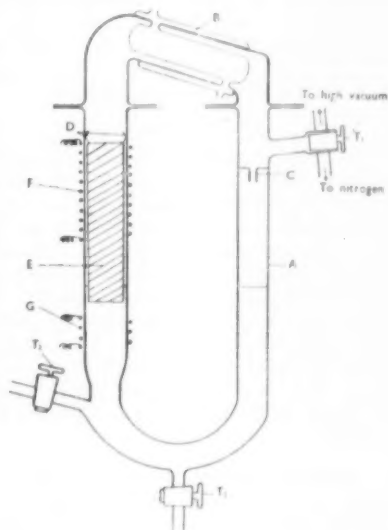


FIG. 1

## Experimental

## EVAPORATION IN NITROGEN

## Apparatus

The Pyrex evaporation apparatus (Fig. 1) was in two parts, an inclined, double-surface condenser B resting with its air-tight, ground flanges on the corresponding flanges of the U-shaped evaporation chamber A. The three-way tap  $T_1$  put A into communication either with a high-vacuum line or with the supply of purified nitrogen. A was connected also with a specially designed still through the tap

$T_2$ . Water, of conductivity quality and free from dissolved oxygen, was prepared in the still, and introduced as required directly into  $A$ .  $A$  was emptied through the tap  $T_2$ . The cloth  $E$  was suspended vertically from the glass hooks  $D$  and dipped 1 cm. below the surface of the water in  $A$ . The resistance-wire heaters  $F$  and  $G$  were fed, through rheostats, from a constant-voltage transformer. The water evaporating from the cloth and condensing in  $B$  collected in drops at  $C$ . From the rate of drop formation, the rate of evaporation could be estimated.

#### *Deoxygenation of the Nitrogen*

"Oxygen-free" nitrogen (British Oxygen Co. Ltd.) was led, past a pressure-release valve, through four scrubbing tubes, 4 ft. long. Three of these contained Fieser's solution (120 g. caustic soda, 2 g. sodium naphthalene- $\beta$ -sulphonate, and 120 g. sodium hydrosulphite per litre); the fourth, caustic soda solution (200 g. caustic soda per litre). It then passed through an electrically heated glass tube containing active copper deposited on kieselguhr<sup>7</sup>, which, it is claimed, can reduce the partial pressure of oxygen in a stream of nitrogen to  $4 \times 10^{-7}$  of the total pressure.

#### *Deoxygenation of the Water*

The apparatus and method have been described by Patrick and Wagner<sup>8</sup>. Distilled water was refluxed for 3 hr. with Wood's metal in an atmosphere of nitrogen, and was then fractionally distilled. It is claimed that the product contains not more than 0.01 ml. oxygen per litre.

#### *Purification of the Cotton*

The starting material, a plain, square-weave cotton cloth ( $2\frac{1}{2}$  oz./sq. yd.) which had previously been desized, was washed, kier-boiled in a 1% soln. of caustic soda for 7 hr. at 20 lb./sq. in. excess pressure, washed, soured, washed, chemicked, soured, and washed. It was then purified by the method described by Bone and Turner<sup>1</sup>, except that the sequence of operations used by them was repeated seven times, and, in the later stages, the cloth was also extracted with chloroform and with alcohol. The cuprammonium fluidity of the purified cloth in 0.5% soln. was 5.8; the copper number 0.05.

#### *Evaporation with Oxygen excluded*

A strip of the purified cloth, 33 cm.  $\times$  5 cm., was put into place in  $A$ , and the whole evaporation apparatus closed and maintained for 72 hr. at a pressure  $< 5 \times 10^{-5}$  mm. of mercury. Purified nitrogen was then admitted, and deoxygenated water, also in a current of the pure nitrogen, was simultaneously distilled into  $A$  until the level rose to 1 cm. above the lower end of the cloth strip. The evaporation of the water, rising by capillary action up the cloth, was accelerated by the heater  $F$ , the extent of the rise thus being limited. By ensuring that the current in  $F$  remained constant during an experiment, the position of the boundary between wet and dry cloth could be kept constant also, within narrow limits, so that the depth of the characteristic boundary region did not exceed

3 mm., and any coloured product was concentrated within this narrow region. On increasing the current in  $F$ , leading to more rapid evaporation, the position of the boundary was lowered; on decreasing the current, the boundary rose. After the evaporation had been started, about 10 min. was needed for the boundary to assume a steady position. A faint brown coloration appeared in the boundary region at the end of 1 hr., and a distinct one at the end of 3 hr. A succession of brown lines could be obtained on the same piece of cloth by periodically increasing the current in  $F$  and so causing the boundary between wet and dry to descend in steps: a line was formed at each position occupied by the boundary. In the reverse procedure, when the current was diminished in steps, only one brown line was obtained, marking the highest position occupied by the boundary. This was because the brown product was transportable in water, and was carried upward as each higher level was established. During these experiments, depending upon the heat input, the rate of evaporation varied between 4 and 8 ml./hr.

At the end of a run, the cloth was removed from the apparatus and washed thoroughly in distilled water, so that practically all the brown substance was extracted. The cloth was then immersed for 7 min. at room temperature in an aqueous solution of methylene blue (0.5 g./litre). Enhanced absorption of the dye occurred at every level where a boundary had existed for any length of time, the intensity of the local staining with the dye being roughly proportional to the length of time the boundary had remained at that level.

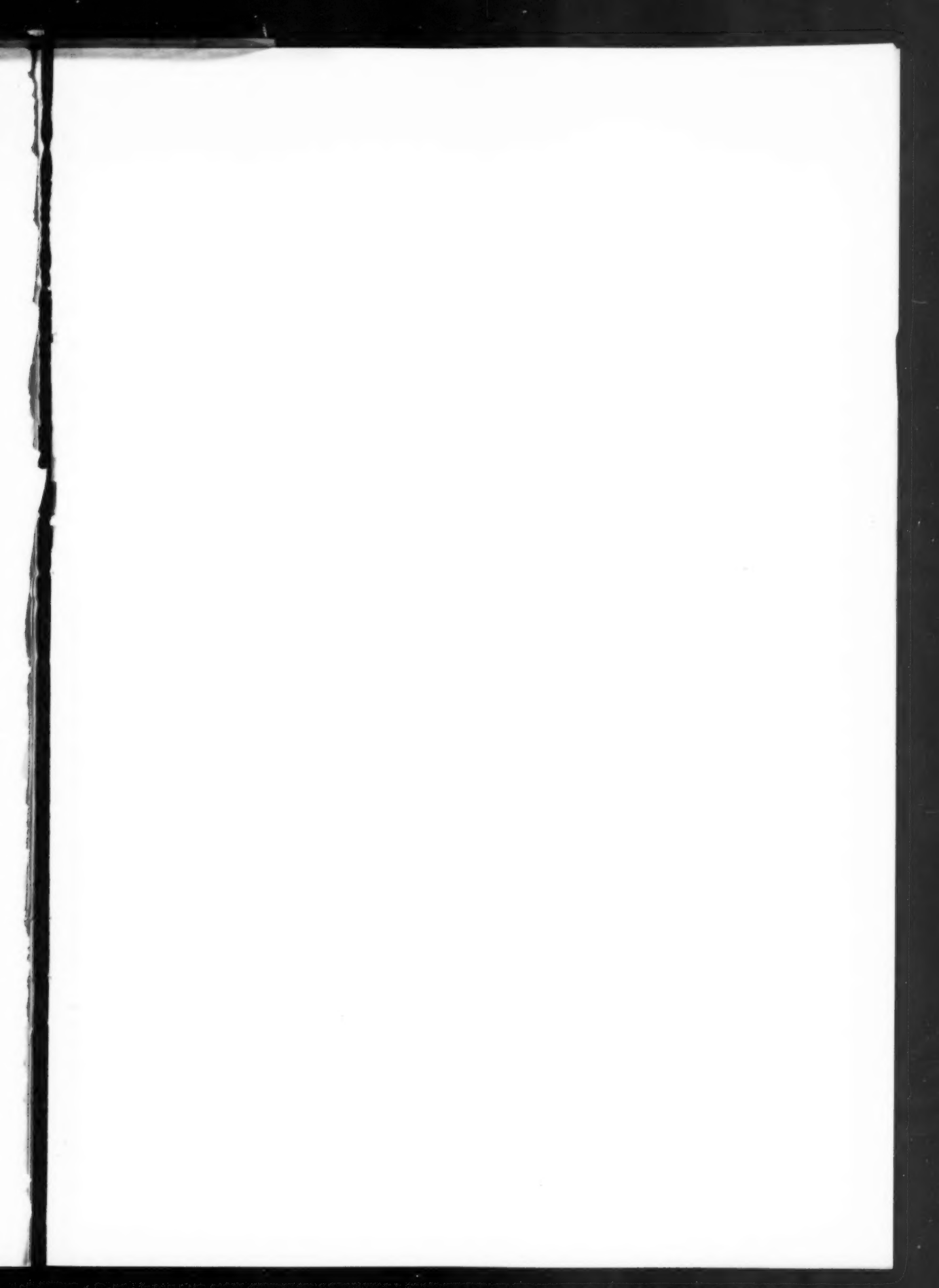
#### EVAPORATION IN VACUO

##### *Apparatus*

The Pyrex apparatus (Fig. 2) consisted of the tube  $a$ , fitted with a ground-glass joint and wound with an electrical resistance heater  $h$ . The lower end of  $a$  was connected with the flat-bottomed flask  $b$ , of 700 ml. capacity and containing a magnetically-operated stirrer. A small tube could be rested on the spoon  $s$ , which could be rotated, transferring the contents of the tube into the flask, without breaking the vacuum in the apparatus. A glass spiral connected  $a$  with the receiving flask  $c$  (250 ml. capacity). A connection from  $b$  led to the vacuum line through a ground-glass joint. By rotation round the axis of this joint, the apparatus could be tilted through a wide angle, and the water in it could be caused to move between  $a$  and  $b$  as desired.

##### *Procedure*

A strip of purified cotton cloth, 16 cm.  $\times$  2.5 cm., was sewn on to a rectangular glass frame, which was then wedged into  $a$ , care being taken that the cloth did not touch the sides of  $a$ . The requisite volume of freshly distilled water was placed in  $b$ , and the apparatus assembled. It was tilted until the water covered the lower end of the strip, which became completely wetted in 10 min. The apparatus was then brought back to its former position, so that all the water returned to  $b$ , the magnetic stirrer was set in motion, and the tap  $t$  was slowly opened to allow evacuation to commence. After evacuation



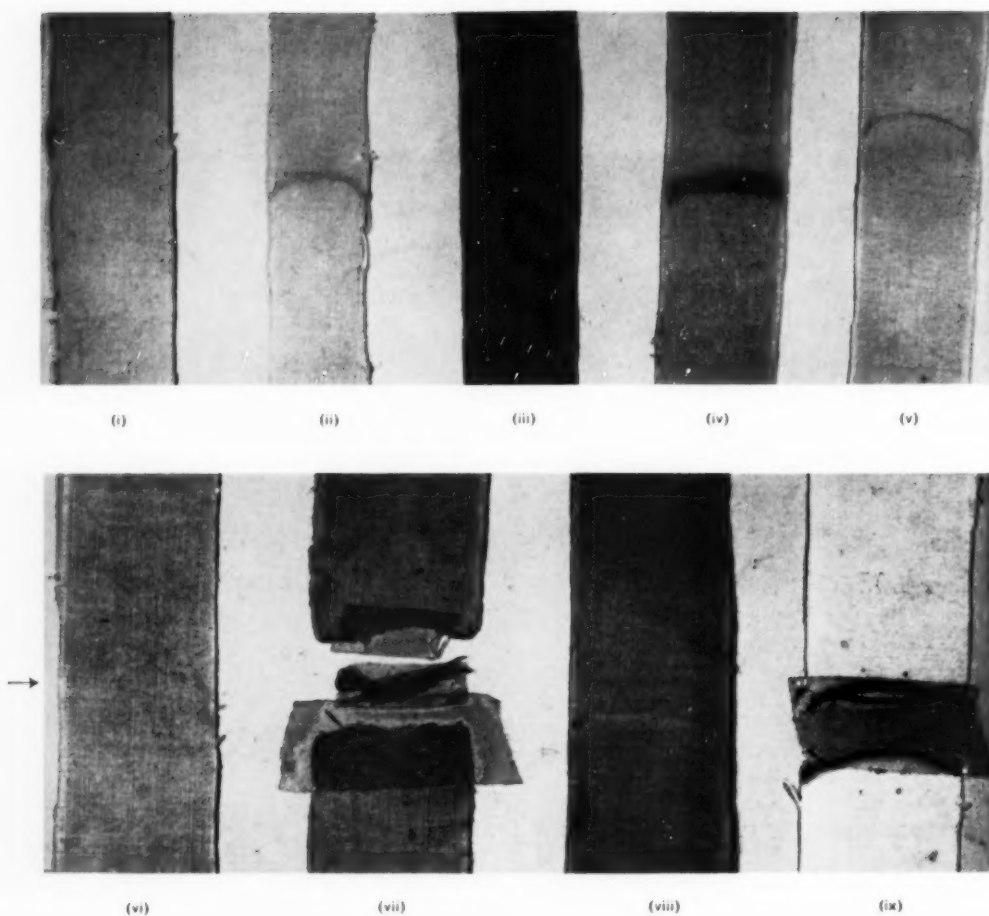


FIG. 3—Cloth from Evaporation Experiments carried out *in vacuo*, after Treatments indicated in Table II

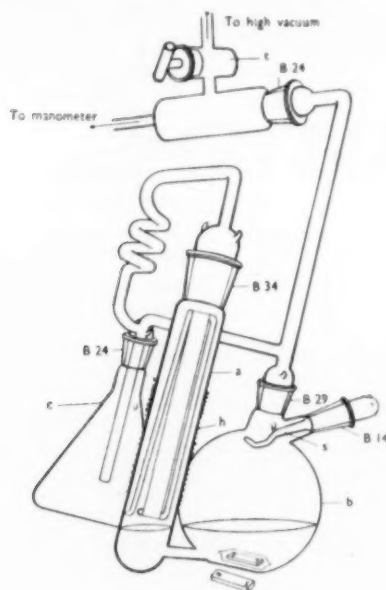


FIG. 2

had continued for about 5 min., the initial ebullition ceased, and some of the water froze. The vacuum line was cut off by closing *t*, the stirrer stopped, and the ice allowed to thaw. The cycle was repeated four times, after which water and cloth were each considered to be free from air.

Tap *t* was at length closed, and the apparatus tilted until the end of the cotton strip dipped about 5 mm. below the surface of the distilled water. The current in *h* was adjusted until the boundary between wet and dry cotton assumed a convenient position. It was found that this boundary could be held stationary, even *in vacuo*, by suitable adjustment of the heater. After the experiment had been started, the apparatus was immersed in an ice-water mixture to a depth which ensured that all the distilled water inside was brought to a temperature near 0°C. This ensured that the temperature difference between the evaporating water on the cloth and the point of condensation in *c* was as large as possible. Control experiments showed that, of the total volume of water evaporating from *a* to *c* in the course of an experiment, 95% evaporated from the cloth strip and less than 5% from the surface of the water supply in *a*.

Experiments were carried out for different lengths of time. At the end of an experiment, the position of the boundary was accurately noted, the apparatus tilted back to its original position, and air admitted through *t*. Alternatively, while the vacuum was still intact, *s* could be operated to drop a thimble containing a strong solution of methylene blue into the water in *b*. After thorough mixing, the apparatus was tilted until the solution of dye completely filled *a*. In this way it was possible to perform the methylene blue test on the cloth pattern without admitting air.

#### COMPARISON OF THE TWO EVAPORATION METHODS

The experiments performed *in vacuo* showed the following advantages over those performed in an atmosphere of nitrogen—

(1) The later apparatus was smaller, more compact, and easier to operate than the earlier one.

(2) The preparatory wetting of the cotton strip, followed by repeated evaporation of the water from it by the action of the vacuum, gave more effective displacement of included and absorbed air than prolonged evacuation of a dry strip under high vacuum. This reduced the length of time required for an experiment from four days to one day.

(3) Water evaporated through the strip, and condensed in *c*, did not again come into contact with the strip.

(4) The risk that oxygen might be continuously introduced into the system, as a trace impurity in the nitrogen, was avoided.

(5) Evaporation from the cloth took place at a much lower temperature (approx. 50–60°C.).

#### ABSORPTION OF METHYLENE BLUE

##### Qualitative

The cloth strips from the evaporation experiments were immersed for 7 min. at room temperature in 200 ml. of methylene blue solution (0.5 g. per litre), and were then washed repeatedly in warm distilled water. Where dyeing was performed in the apparatus by releasing the glass thimble into *b* (Fig. 2), the strength of the resultant dye solution was  $1.9 \times 10^{-5}$  M., and the time of dyeing was 16 hr.

In some experiments, the strips from the apparatus were dyed in a Marney machine with 200 ml. of a solution of pure Chlorazol Sky Blue FF (ICI) (1 g./litre) for 25 min. at 75°C.

##### Quantitative

Commercial zinc-free methylene blue chloride was twice crystallised from water, and dried to constant weight at 105°C. The purity was 94.5–94.7% (dichromate method<sup>9</sup>) or 93.0–93.5% ( titanous chloride method<sup>10</sup>), the latter values being adopted in calculations. Methylene blue stock solution, buffered at pH 8, was prepared according to the method of Davidson<sup>11</sup>. By diluting this solution with different proportions of 0.1 N. acetic acid, a number of standard solutions for calibrating the Spekker photoelectric absorptiometer were obtained.

Portions of the cotton strip, 2.5 cm.  $\times$  1 cm., were cut in duplicate from the wet and from the dry regions together with a portion cut to include the boundary region. Each portion was weighed and was immersed in a separate volume (10 ml.) of methylene blue solution ( $1.3 \times 10^{-5}$  M., pH 8) at  $22.5 \pm 0.2^\circ\text{C}$ . for 18 hr. Five millilitres of dye solution was then taken and mixed with 5 ml. of 0.1 N. acetic acid, the dye content of the mixture being at once estimated with the Spekker.

#### Results

##### EXPERIMENTS IN NITROGEN

In these experiments the reproducibility of the results was consistently poor, and only the most

TABLE I  
Evaporation of Distilled Water in Nitrogen

Experiment No.	Pressure in Preliminary Exhaustion (mm. Hg)	Heating Current (amp.)	Gas present	Duration of Evaporation (hr.)	Appearance
1	10 <sup>-4</sup>	2.3	Purified N <sub>2</sub>	11	Faint brown line
1 (a)	—	2.35	Air	3	Brown line formed
				6	Intense brown line
2	10 <sup>-4</sup>	2.25	Purified N <sub>2</sub>	11	Intense brown line
				16	Little further change
2 (a)	—	2.25	Air	11	Intense brown line
				16	Little further change

tentative conclusions are permissible. This feature is brought out in Table I, which includes sample results typical of a much larger number obtained over a period of several months. Some of the experimental difficulties were undoubtedly due to the necessity for the scrupulous removal of oxygen from the beginning, and its continued exclusion during the four days required for a single complete experiment. Even when there was reasonable certainty that these two conditions had been approached as closely as the design of the apparatus would allow, the inconsistent results still appeared. Apart from the fact that oxygen had been more completely removed from the evaporation system, there was a further difference between the present apparatus and that of Bone and Turner. The maintenance of the brown line at a convenient height from the lower end of the cloth required that the surface temperature should be much higher. As far as could be observed, this had no special influence on the formation of the line, although the final properties of the brown material, in particular its solubility, may have been modified to some extent<sup>6</sup>. This point was not investigated very closely.

From the experiments which proceeded satisfactorily, the following observations of Bone and Turner were substantially confirmed—

- (1) Evaporation of distilled water from a fabric, under the conditions defined, resulted in a sharp boundary between wet and dry regions.
- (2) Modification of the cellulose took place at this boundary with the production of a brown, water-transportable, fluorescent reaction product.
- (3) After extraction of this brown material, there was evidence of modification of the residual cellulose at the place where it had been formed. The methylene blue absorption was markedly greater, and there was evidence of a slight local increase in the cuprammonium fluidity as compared with that of the wet or the dry regions of the cloth.
- (4) Periodic lowering of the evaporation level in the same cloth sample during the same experiment gave rise to a series of fresh brown lines of undiminished intensity, although, at each lowering, the area of wet cloth from which water could evaporate was correspondingly reduced.

Observation (4) seems to be conclusive evidence that the brown line does not represent merely a concentration at the boundary of non-cellulosic material present originally in the cloth sample or formed during the experiment in the whole of the

wet region. This conclusion agrees with that expressed by Appel *et al.*<sup>6</sup>.

Because the cellulose appears to be modified only at the boundary between wet and dry cloth, free and intimate access of atmospheric oxygen to wet cellulose seems to be required. However, comparison of the results obtained when evaporation takes place in nitrogen with those obtained when air is deliberately admitted leads to no definite conclusion. In Experiments 1 and 1(a) (Table I) the colour of the brown line formed in nitrogen was less intense than that of the line formed in air. In Experiments 2 and 2(a) the corresponding colorations were about the same. If oxygen is indeed necessary before the brown line can be formed, it seems that only very minute amounts are required.

One observation is particularly interesting. An experiment in nitrogen had been proceeding for several hours, and a faint coloration only had been formed. Air was then admitted, whereupon the coloration intensified very rapidly. As some of the later experiments *in vacuo* also suggest, this observation leads to a tentative conclusion that evaporation in the absence of oxygen can cause a modification of the cellulose, although the products of the reaction are colourless. This modified cellulose requires oxygen for the brown product to be formed.

In the present experiments, the brown, water-soluble reaction product reduced ammoniacal silver nitrate solution, but had no action on a sensitive Schiff's reagent. Attempts to identify simple reducing sugars in the brown product by paper chromatography were not successful. Recently the presence of small amounts of uronic acids has been reported<sup>6</sup>.

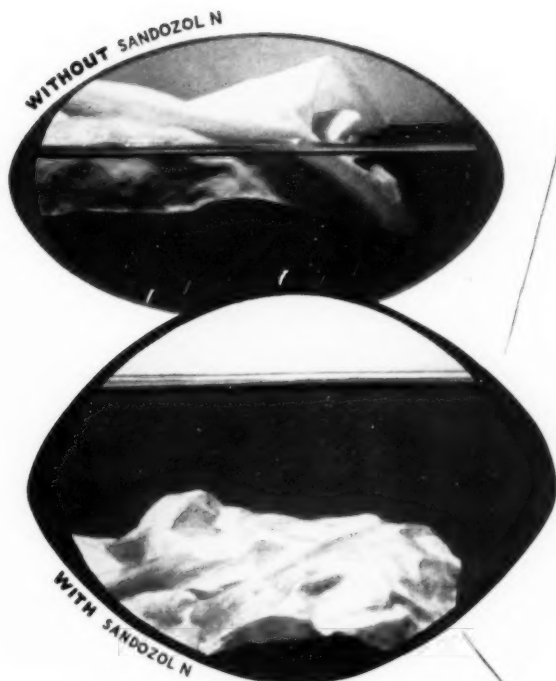
#### EXPERIMENTS IN VACUO

The greater convenience and reliability of this method, as compared with the one just described, enabled a greater number of significant results to be obtained, and allowed an extension of the experiments to include the evaporation of dilute solutions of acid and of alkali through the cloth. Typical results have been collected and set out in Table II.

#### Distilled Water

All experiments were carried out at a pressure of 7 mm. Hg, and the current through the heater was 0.18 amp. A sharply defined boundary between

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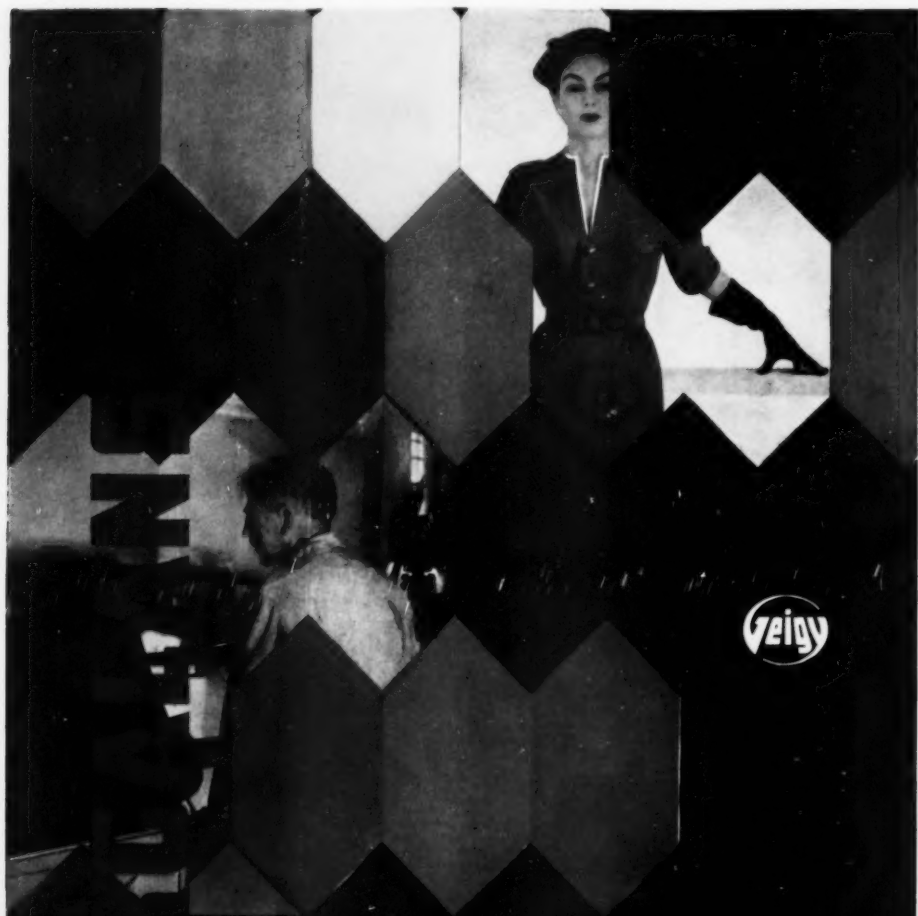
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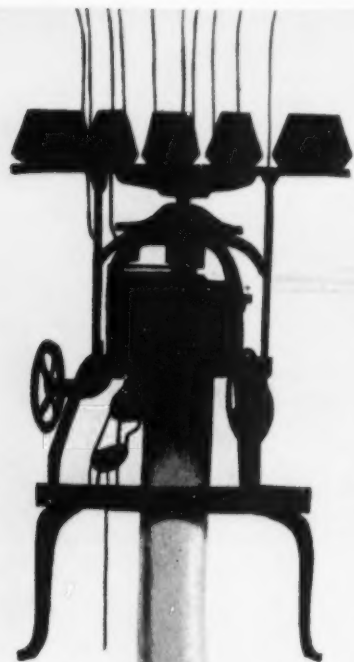
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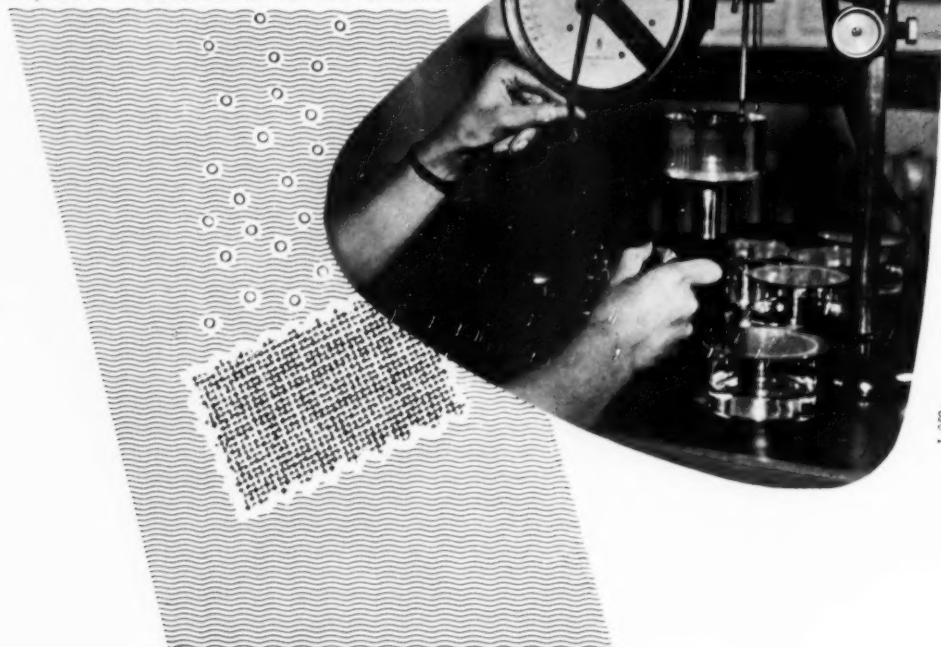
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TABLE II  
 Evaporation *in vacuo*

No. in Fig. 3	Duration of Evaporation (hr.)	Water Evaporated (ml.)	Appearance at Boundary before Staining	Dye Absorption at Boundary*	Further Treatment after Staining	Remarks
DISTILLED WATER						
(i)	5.25	14.5	No discoloration	Enhanced	—	—
(ii)	18.75	43.0	No discoloration	Enhanced	—	—
(iii)	7.0	22.0	No discoloration	Unchanged †	—	—
0.001 M. SODIUM CARBONATE						
(iv)	6.5	23.0	Slight discoloration	Greatly enhanced	—	—
(v)	6.0	23.0	No discoloration	Greatly enhanced	Wash with distilled water	Intense narrow line remains
(vi)	5.0	18.0	Slight discoloration	Diminished ‡	—	—
0.001 M. HYDROCHLORIC ACID						
(vii)	6.0	23.0	Insol. yellow-brown discoloration	Diminished	Dil. H <sub>2</sub> O <sub>2</sub> ; then again stained*	Dye absorption greatly enhanced; tensile strength diminished
(viii)	4.5	18.0	Sol. yellow discoloration	Diminished	—	—
(ix)	10.0	36.0	Sol. yellow discoloration	Unstained	Air admitted after 10 hr.	Intense dark brown line; tensile strength diminished

\* Stained with methylene blue (1 g./litre) for 7 min. in the cold

† Dyed with Chlorazol Sky Blue FF (1 g./litre) for 25 min. at 75°C.

‡ Specimen washed thoroughly with distilled water before staining

wet and dry cloth could be set up and could be maintained in one position during the complete run of the experiment. No coloration was observed at the boundary for periods up to 19 hr. For the first time it has been possible to carry out a lengthy evaporation without the formation of a brown line, and since the purification of cloth and water has followed the same course as in previous experiments, it seems highly probable that adequate exclusion of gaseous oxygen has at length been attained. However, on staining, the position of the boundary was still marked by an enhanced absorption of methylene blue, the preferential absorption being roughly proportional to the duration of an experiment (Fig. 3 (i) and (ii)). This suggests that modification of cellulose at the boundary still accompanies the evaporation of water, and that the modification product is acidic. A product deriving truly from cellulose is unlikely to be made more acidic except by the formation of -COOH groups. It should be noted that differential absorption in the pattern could not be obtained on staining with the direct cotton dye, Chlorazol Sky Blue FF (Fig. 3 (iii)).

#### Sodium Carbonate Solution

The concentration of the carbonate solution was 0.001 M., and again the pressure in the apparatus was 7 mm. Hg and the heating current 0.18 amp. If the modification of the cellulose at the boundary in these experiments is due to oxidation, the presence of alkali should encourage the reaction, and very minute traces of residual air, which may be insufficient to cause any change with distilled

water alone, may here give some coloration. The typical results given in Table II are, on the whole, very similar to those with distilled water, with some variation in subsidiary details. In some experiments, a faint irregular coloration at the boundary was observed, and this spread over a band 0.2–0.5 cm. wide. The local increase in methylene blue absorption at the boundary was much greater in experiments with dilute sodium carbonate (Fig. 3 (iv)). Much of the product apparently responsible for the high dye absorption was soluble, since, after extraction with water, the enhancement of dye absorption was much diminished (Fig. 3 (v) and (vi)). With Chlorazol Sky Blue FF, there was again no evidence of local change in dye absorption.

#### Dilute Hydrochloric Acid

The concentration of the acid was 0.001 M., the working pressure in the apparatus was 7 mm. Hg, and the heater current was 0.18 amp.; the results of typical experiments are again shown in Table II. Colour developed rapidly in a band a few millimetres above the boundary between wet and dry cloth. In 2–3 hr. a yellow material, soluble in water, was formed, but on extension of the time of the experiment, the brown or yellow coloration could not be removed with water. Admission of air greatly accelerated the colour change from yellow to brown (Fig. 3 (ix)). Methylene blue absorption at the boundary region was reduced as compared with the rest of the cloth, and this happened whether the samples were washed before staining or not (Fig. 3 (viii)). Some samples, after being stained with methylene blue, were

immersed in a dilute solution of hydrogen peroxide. A repetition of the staining showed a greatly increased local absorption of methylene blue (Fig. 3 (vii)). In all the experiments, the cotton at the boundary region was more tender than the rest of the pattern (Fig. 3 (vii) and (ix)). These results are consistent with the modification of the cellulose at the boundary by a hydrolytic process, giving rise to material which can then be oxidised by hydrogen peroxide to products which are rich in  $-\text{COOH}$  groups.

#### METHYLENE BLUE ABSORPTION IN VACUO

The results obtained in experiments with distilled water or dilute sodium carbonate solution for the evaporation were essentially the same whether the cloth was removed from the apparatus before being stained with methylene blue or whether the staining took place in the apparatus before the vacuum was broken. This showed that modification of the cellulose, to produce material at the boundary with a high affinity for methylene blue, actually occurred during the evaporation of the water and was not delayed until air had been admitted.

In the experiments with dilute alkali and dilute acid solutions, staining within the apparatus means necessarily that the dye solution is applied at different pH values in the different experiments. This may have been responsible in part for the new behaviour observed with dilute hydrochloric acid as the evaporating liquid. Here the original line of the boundary showed a very faintly enhanced coloration with the basic dye, but a second, more diffuse line, approx. 2 mm. higher, showed decreased dye absorption. This effect was not further investigated, but it may be due, as suggested later, to a local increase in the concentration of the acid near the boundary region.

#### NON-POLAR LIQUIDS

By the same procedure as with water or the aqueous solutions, spectroscopically pure non-polar liquids—*n*-hexane and cyclohexane—were evaporated through the cloth. These liquids possessed convenient rates of evaporation in the vacuum apparatus. No significant evidence of modification of the cellulose could be obtained.

#### QUANTITATIVE DETERMINATIONS OF METHYLENE BLUE ABSORPTION

Since the weight of material at the boundary region showing enhanced methylene blue absorption was so small, and was not clearly defined until the actual staining procedure had been performed, the determinations were made as follows—After evaporation, the strip was removed from the apparatus and dried. Portions of different weight were cut from the parts of the strip which did not include the boundary region. Each portion was weighed accurately, and then the staining performed in the standard manner, the dye absorption being determined colorimetrically. From the results a straight-line curve relating dye absorption with weight of cotton was obtained. This represented the normal dye absorption of the cloth. A sample

was now cut to include the boundary region and weighed, and the dye absorption was determined. From the curve, the weight of dye absorbed by the same weight of normal cloth was ascertained, and this value, subtracted from the observed value, gave directly the enhanced absorption due to the presence of the boundary region. During staining, the exact limits of the region of enhanced dye absorption had been revealed. The cloth corresponding to this region was now carefully cut out from the larger sample and the weight obtained. From this figure, the enhanced dye absorption due to modification of the cellulose could be expressed in terms of the weight of cotton on which modification had taken place. Typical results are given in Table III. In experiments with distilled water,

TABLE III  
Enhanced Methylene Blue Absorption for Distilled Water Experiments

Experiment No.	Wt. of Cotton carrying Modified Cellulose (a) (mg.)	Enhanced Methylene Blue Absorption due to (a) (micromoles)
1	3.9	0.8
2	3.5	0.5
3	3.9	0.7

there was a preferential dye absorption at the boundary of  $1.75 \times 10^{-4}$  mole of dye for each gram of cotton (the mean of the three results in Table III). The value for experiments with dilute sodium carbonate was  $3.5 \times 10^{-4}$  mole per gram. In the experiments with dilute acid, the results were inconclusive because of the formation of two separate lines, each with distinct absorption properties. The net result, obtained from cut samples which necessarily included both lines, was of diminished methylene blue absorption.

#### Discussion

The most significant result in the present work is the evidence that, with more thorough removal and exclusion of air from the evaporation system, the formation of a brown coloration at the boundary is inhibited, but that localised modification of the cellulose has not been suppressed. Even at this stage of oxygen exclusion, however, the local modification product is still acidic in properties. If this acidity is organic in origin, it is difficult to imagine that it is due to anything other than  $-\text{COOH}$ , and there is thus evidence of a process of oxidation in spite of the most strenuous attempts to exclude oxygen from the system. The second problem to be solved is why the modification of the cellulose appears to be so much more pronounced only at the boundary between wet and dry cellulose. Taking the experimental evidence in the present study as of absolute significance, the formation of a brown line is the only part of the total phenomenon which can now be connected with oxidation. Any other modification of cellulose to give material of lower molecular weight should thus be hydrolytic. In the original paper of Bone and Turner<sup>1</sup>, a number of suggestions were made to explain why modification is restricted to the boundary area. There is no need to repeat these

in detail, and the original paper should be consulted. They may be referred to as—(a) an advancing and receding tide mechanism\*, (b) concentration of surface charge mechanism, and (c) autohydrolysis of cellulose from carboxyl groups already present, or formed in minimal quantities by traces of residual oxygen. Since the first paper was published, Davidson and Standing<sup>13</sup> have given an account of the autohydrolysis of cellulose originating from the  $-\text{COOH}$  groups normally present. None of the evidence in the present study disproves these suggestions, but none of it is able to allow a selection to be made of a most probable explanation from among the competing theories.

Each of the hypotheses—(a), (b), and (c)—leads to the idea of a localised hydrolysis of cellulose at the boundary region. The experiments in a vacuum appear to have relegated oxidation processes to the secondary production of the brown material. How, then, can the appearance of  $-\text{COOH}$  groups at the boundary be explained? The suggestion is now made that these are formed from terminal  $-\text{CHO}$  groups, produced by hydrolysis, through a Cannizzaro reaction. This is an unusual suggestion because it implies that  $-\text{COOH}$  and  $-\text{CH}_2\text{OH}$  are formed from two  $-\text{CHO}$  groups, in the experiments with distilled water, at a much lower pH than that at which the reaction is normally found to take place<sup>14</sup>. It seems, however, to be the only suggestion which can explain why  $-\text{COOH}$  groups can be formed in the absence of a source of oxygen external to the cellulose. The suggestion receives some support from the experiments with dilute sodium carbonate, where the conditions are more favourable to the Cannizzaro reaction. Here, in fact, there is a more copious formation of products, both soluble and insoluble, with an enhanced absorption capacity for methylene blue.

A tentative suggestion, requiring more work before it can be supported with confidence, is put forward to explain the effects observed during the evaporation of dilute acid through the cellulose in a vacuum. With a relatively large hydrogen ion concentration, hydrolysis of the cellulose is favoured. There are, it has been seen, two distinct lines in the vicinity of the evaporation level, the lower one colourless with a slightly enhanced methylene blue absorption, the upper one coloured yellow. It is at this latter line that most tendering

of the cellulose occurs. The methylene blue absorption is below normal. It is suggested that the colourless lower line corresponds to the effect observed in the evaporation of water alone, and that the upper one is caused by a local increase in concentration of the hydrochloric acid, leading ultimately to a composition identical with that of the constant-boiling mixture, which possesses a higher energy of evaporation than that of distilled water alone. This would give a very acid region in which marked hydrolysis would occur, probably with caramelisation of the primary hydrolysis products. The low methylene blue absorption of this region after washing may be accounted for by the fact that conditions do not favour the disproportionation of the aldehydic hydrolysis products, and that basic dye will not be taken up until these groups have been converted into carboxyl by a subsequent treatment with hydrogen peroxide.

\* \* \*

We should like to thank Mr. F. Scholesfield, Dr. J. H. Baxendale, and Mr. A. Johnson for interest and for helpful advice during the course of the work; Mr. H. Stewart for care and patience in the construction of some of the apparatus; and the directors of the Bradford Dyers' Association Ltd. for the award of research fellowship which enabled one of us (G. W. M.) to take part in the work.

DEPARTMENT OF TEXTILE CHEMISTRY  
COLLEGE OF TECHNOLOGY  
UNIVERSITY OF MANCHESTER

(Received 11th March 1953)

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- <sup>12</sup> Hermans, *Contribution to the Physics of Cellulose Fibres* (Amsterdam and New York: Elsevier Publishing Co. 1946), p. 22.
- <sup>13</sup> Davidson and Standing, *J. Textile Inst.*, **42**, T 141 (1951).
- <sup>14</sup> Hermans, *Physics and Chemistry of Cellulose Fibres* (Amsterdam and New York: Elsevier Publishing Co. 1949), p. 136.

\* An example has been given by Hermans and Vermaas<sup>12</sup> of the sharp differentiation of swollen and unswollen cellulose within a fibre as liquid water advances into it from the periphery.

## Notes

### Meetings of Council and Committees September

Council—9th  
Finance—9th and 15th  
Publications—15th  
*Review of Textile Progress*—11th  
Dyers' Co. Research Medal—8th  
Joint Conference with Textile Institute—17th  
Perkin Centenary—29th

### Centuries-old Irish Tweed

The National Museum of Science and Art, Dublin, has recently received pieces of a herring-bone tweed found on the skeleton of a man at a depth of six feet in a peat bog near Newbridge, Kildare. The cloth is strong enough to be handled and has a clean pattern, though it has been coloured brown by the peat. Mr. A. Lucas, the curator of the museum, believes that the cloth may date from medieval times, and is engaged in work to establish its precise date and, so far as possible, the processes used in making it. c o c

## New Books and Publications

### Neueste Fortschritte und Verfahren in der chemischen Technologie der Textilfasern

#### Zweiter Teil

### Neue Verfahren in der Technik der chemischen Veredlung der Textilfasern Hilfsmittel in der Textilindustrie

By Louis Diserens. Vol. II, pp. xi + 916.  
Basle: Verlag Birkhäuser. [2nd edition]  
1953. Price, 118.50 Swiss francs.

This book, the second volume of Part II of Dr. Diserens's notable work on textile bleaching, colouring, and finishing, deals with the constitution and the chemical properties of a large number of commercial products employed in textile processing. The matter is arranged in five chapters with a patent index and an alphabetical list of the commercial products mentioned in the text. In addition there are a number of tables, which group products together according to chemical constitution. The first chapter (Chapter VII) deals with starches, gums, synthetic resins, rubber, cellulose derivatives, and silicones. The second chapter, the largest section of the book, is concerned with surface-active agents. Chapter IX covers miscellaneous topics such as optical bleaching agents and antistatic agents, whilst the last two chapters deal with antiseptics and moth-proofing agents respectively. The book is to be followed by a further volume dealing with durable finishing agents, water-repellents, crease-resist agents, flameproofing, and so on, and its publication will complete the task which Dr. Diserens has set himself.

The present volume covers old and new ground, the survey of existing products being comprehensive, and the literature references very full. It will therefore prove of value to technologists in the user industries who wish to have at hand a review of the chemistry of textile auxiliaries.

It is a task of great difficulty to classify the large numbers of products mentioned, and to assign to them their correct constitutions. The author can therefore be forgiven a number of the errors which appear in the text.

On p. 418 there is a curious error which has appeared in other surveys of surface-active agents; it refers to the constitution of Fixanol. This product is cetylpyridinium bromide, not octadecylpyridinium sulphate.

To list a number of other points which might receive attention in later editions of the book, Humectol CX and Dismulgan V are assigned different formulæ on p. 326. Both are diisobutylamine derivatives, whilst Dismulgan IV is the ethylaniline derivative. On p. 347, it is implied that cetyl chloride is a fatty acid chloride. Nekal A is almost certainly a diisopropylnaphthalene derivative, not the monopropyl compound as suggested on p. 386. The length of the alkyl chain in Naccenol NR is probably in the C<sub>14</sub> range, and not C<sub>16</sub>-C<sub>18</sub> as suggested on pp. 388-389. Different methods of representing the pyridine ring are adopted on pp. 419 and 420, and this might be amended in the interests of consistency. It is questionable whether Igepal NA and Peregol OK have the constitutions assigned to them on pp. 445 and 446 respectively.

The book is beautifully printed and produced, and represents a capable attack on a difficult subject. J. R. F. JACKSON

### Die I.G. Farbenindustrie Aktiengesellschaft Ihre Entstehung, Entwicklung und Bedeutung

By Fritz ter Meer. Pp. 123. Düsseldorf: Econ-Verlag G.m.b.H., 1953. Price, DM. 4.80.

Dr. ter Meer can be sincerely congratulated on writing this book, and it is amazing how he has been able to give a mass of information within the confines of this small publication. The book measures 7½ by 4½ in., and has about 115 pages. He discusses the German dye industry and its allied industries in sufficient detail to point out the moral of the story he wishes to tell. His story begins in 1856, and discusses the early years of the industry in Germany, which led finally to the creation of the I.G. His story is divided into chapters with subheadings, and all aspects (chemical and commercial) of the I.G. are touched upon. He has avoided the arrogance so common in

German publications between the wars, and all he writes is eminently readable. Of course, he writes with justifiable pride in German achievements in research and production. No doubt his book was published mainly for German consumption, but, owing to its excellence, it will have a much wider public.

Fritz ter Meer is an eminent chemist and administrator, whose works were well known for their production of the cationic range of dyes before the I.G. was formed.

The book, which is pleasantly if simply bound, is a "must" to be read by all interested in administration, be it engineering, chemical, technical, or commercial. It is not intended for those specialising in necessary but narrower fields. Most organisational aspects of the I.G. are discussed frankly, and figures and graphs are quoted when required.

Most unfortunately, ter Meer, of all people, makes a dreadful "howler" in the opening sentence, in that he gives William Henry Perkin the Christian name of "Thomas". This mistake is more unfortunate than would be calling ter Meer "Carl" or Duisberg "Fritz". But one is glad that otherwise he quotes only surnames, as we do in Great Britain, when writing about his colleagues. After all, it is frightening to British eyes to read that "Geheimer Kommerzienrat Professor Doktor Soundso" did this or that.

He tends not to admit that a strong chemical industry is essential to a Great Power other than Germany, and one is surprised that he laments the production of dyes and organic chemicals in other countries. No doubt that this type of thought and the principles of autarky led to the formation of the I.G., and ter Meer discusses this quite fully. He points out, though, that as early as 1903 steps were in view to form combined interests, since Duisberg (Baeyer) visited the U.S.A., and there saw the creation of trusts moving rapidly.

One or two arguments in the book may irritate readers to some extent, but it would be churlish to underline them in this review. There is so much of interest and value, and lessons in it which we would do well to study.

ter Meer discusses fully and in sufficient detail for the purpose of his book the formation of contracts, of syndicates both national and international, and finally of world-wide cartels. The comments make most interesting reading. Some of the chapters, or rather subsections, are enlivened by remarks made by people of the eminence of Bosch and Duisberg, and shows the totally different outlooks and temperaments of these two great men.

It would require a lengthy review to show the full value of this book. In the opinion of the reviewer it should be translated into English, and, please, with an index.

H. A. BRASSARD

### Adolf von Baeyer

1835-1917

By Karl Schmorl. Pp. ix + 214. Stuttgart: Wissenschaftliche Verlagsgesellschaft m.b.H., 1952. Price, DM. 12.50.

This book is Volume 10 in the series *Grosse Naturforscher* ("Great Scientists") edited by

Dr. H. W. Frickhinger, and it is devoted to the life and work of Adolf von Baeyer, one of the greatest chemists of all time. Among his predecessors in the series are Humphrey Davy, Berzelius, von Helmholtz, Bunsen, Wöhler, and Ehrlich, names which will give some idea of the status of Baeyer in scientific setting. Most chemists will remember Baeyer for his discovery of the constitution of natural indigo and its synthetic preparation, probably the greatest achievement in colour chemistry. Although twenty-nine short biographical notices, including the Memorial Lecture by Perkin (*J. C. S.*, 123, 1520 (1923)) have appeared in all, it is remarkable that no more comprehensive treatment of Baeyer's life has been forthcoming since his death in 1917. This volume now remedies a serious defect, since Baeyer was in a special sense a link between the nineteenth and the twentieth centuries, i.e. between the era of classical structural chemistry and that of electronics.

The book deals with Baeyer's life in chronological order, giving the usual biographical details of parentage, youth, adolescence, and university career in Berlin. A brief description is given of life in Berlin around 1850, where we learn that among Baeyer's friends was Ernst Haeckel, who later became the famous biologist and author of *The Riddle of the Universe*. Chemistry, however, received scant attention in Berlin at this period, so we find Baeyer migrating to the world-famous laboratory of Bunsen in Heidelberg, where his companions included Roscoe, Lothar Meyer (the co-discoverer with Mendeleev of the Periodic Law), and Beilstein. It was in Heidelberg that he became closely associated with Kekulé, and Baeyer's bent for structural chemistry must be largely attributed to the influence of this great molecular architect. Separate sections of the book give vivid descriptions of Baeyer's subsequent activities in Ghent, Berlin, Strasbourg, and finally Munich, where he succeeded Liebig in 1875 and worked continuously for the next forty years until his retirement in 1915. A bibliography is given of Baeyer's most important publications, and these exceed 300. Among his pupils appear most of the great organic chemists since colour chemistry made its advent with Perkin's mauve in 1856, including E. and O. Fischer, Graebe, Liebermann, Friedländer, Curtius, R. and V. Meyer, Einhorn, Bamberger, W. H. Perkin jun., Buchner, Rufe, Thiele, Willstätter, Dimroth, Wieland, and Meisenheimer. Among prominent industrialists we note Caro, Schraube, Wieler, von Weinberg, C. Duisberg, Villiger, Hepp, and ter Meer; and among foreign chemists there appear the names of Goldschmidt, Hollemann, Nef, Angeli, W. A. Noyes, Ipatiev, Walden, and Gomberg.

Separate sections deal with the award to Baeyer of the Nobel Prize in 1905 for his work on dyes and hydroaromatic compounds, life in the university and city of Munich in Baeyer's time, his social activities and his idiosyncrasies, family life in Starnberg, Baeyer as an artist, and Baeyer as an examiner, where we learn that the pharmacists had given the nickname of "Bleikammer" to the examination hall. Finally come sections on his

last twelve years, death in 1917, and subsequent memorials. His greatest pupil, E. Fischer, wrote that Baeyer since the time of Liebig had been the greatest teacher of chemistry in Germany and probably in the whole world.

Anyone who wishes to know the details of the life of a genius who lived for creative work only, will find them in this book. To the reviewer, the reading of the book has been a great inspiration, which he is confident will be the like reward of other readers.

H. H. HODGSON

### **Introduction to Cleaning Technology for Dry-cleaners and Launderers**

By Ernest Albinson. Pp. 111. London: Trader Publishing Co. Ltd. 1952. Price, 10s. 6d.

The dry cleaning industry owes a debt of gratitude to Mr. Albinson for the work he has done in helping to inaugurate the training classes for dry-cleaning operatives. These classes have raised the standard of skill in the industry, and have been of real benefit to the trade.

The book under review is a further attempt to implement the aims of such training classes, and is correctly titled an Introduction. An attempt to state the elements of chemistry in ten pages is ambitious, but fails in its purpose. If the readers of this book have studied chemistry, they will not need such a review; and if they have not studied the subject, they will not benefit greatly from such a cursory treatment of the subject.

The remaining chapters, on Textile Fibres, Solvents, Wet-cleaning Agents, Detergency, Dry-cleaning Soaps, Mothproofing, Flameproofing, and Waterproofing, contain useful information and should be of value to those firms which do not employ technical staffs.

The illustrations and diagrams are very well reproduced, and the layout of the book is excellent.

A. BREARE

### **ASTM Standards on Soaps and other Detergents**

(with related information)

Prepared by A.S.T.M. Committee D-12 on Soaps and other Detergents. Pp. viii + 537-685. Philadelphia: American Society for Testing Materials. Price, paper, \$2.25.

This is a reprint of part of the 1952 *Book of ASTM Standards* (Part 7) together with some additional items of particular interest to manufacturers, consumers, and analysts of detergents and allied products.

Tentative and Adopted Specifications for 17 soap products and 9 alkalis (described as "alkaline detergents") are included, many being also approved by the American Standards Association. Analytical methods for these, for soap products containing synthetic detergents, for sulphated and sulphonated oils, and for metal-cleaning compositions are given. There is a standard corrosion test for aluminium cleaners, and a list of definitions adopted as standard.

In appendixes are given proposed methods for determination of surface and interfacial tensions (du Nouy methods), and proposed tests for rinsing,

buffering action, and corrosion of other metal cleaners.

The book is clearly printed and remarkably free from errors. One cannot, however, reconcile a maximum permitted  $\text{Na}_2\text{CO}_3$  content of 9.0% in sodium bicarbonate with a minimum  $\text{NaHCO}_3$  content of 96.0%.

As in previous A.S.T.M. Standards, "detergent" continues to be defined as "a composition which cleans". Sodium bicarbonate, borax, and seven other named types of alkali are thus labelled "alkaline detergents", which are defined as water-soluble inorganic alkalis or alkaline salts having detergent properties, but containing no soap or synthetics. The first definition is vague, and so much broader than commonly accepted usage that scouring powders, acids, polishes, solvents, and even water would readily qualify without absurdity, while the second appears to be so restrictive that a mixture of soap or synthetic detergent and alkali is not an alkaline detergent.

In a book of this nature, much is necessarily repetitive. It is not the object of the book to fill in the omissions where no standards have yet been proposed. Thus, analytical methods for synthetic detergents are confined to sulphated and sulphonated oils, and other synthetic detergents are calculated by difference methods. No mention is made of small amounts of surface-active agents normally remaining with alcohol-insoluble residues; nor are carboxymethyl cellulose and methods for oxidising agents mentioned. The only phosphates recognised are trisodium phosphate and tetrasodium pyrophosphate, for which both standards and analytical methods are given.

In this country, the book will be of considerable use to analysts, who will find therein accepted standard methods for products and their constituents covering a considerably wider range than at present covered by British Standards. Dimensioned diagrams of several pieces of apparatus are included. An index would have been an asset.

D. F. B. KEVAN

### **Surface Finishing of Aluminium and its Alloys**

(A.D.A. Information Bulletin No. 13)

### **Anodic Oxidation of Aluminium and its Alloys**

(A.D.A. Information Bulletin No. 14)

No. 13: Pp. 43, [1953], No. 14: Pp. 64, [1949].

London: The Aluminium Development Association. Price, paper, 2s. 6d. each.

Bulletin No. 13 details the many processes whereby a desirable finish may be given to an aluminium or aluminium alloy surface, except for the anodising process, which is touched on only lightly in this book (e.g. as a pretreatment process).

After general details of the methods of pretreatment (cleaning and degreasing) come descriptions of mechanical processes for creating a finish (polishing, sand-blasting, hammered effects, etc.) chemical processes, electroplating, and, finally, paint finishes.

This booklet has been well and pleasingly produced with several good black-and-white illustrations, and the instructions for obtaining the various effects are lucid and easy to follow.

However, as dyers and colourists we must necessarily be more interested in Bulletin No. 14. It is obvious that the Aluminium Development Association has taken great trouble to ensure that every aspect of anodising is properly covered, from the history of anodic oxidation, through all the usual and less common coloured effects, to, finally, detailed tests to determine the quality of the finished articles.

The foreword describes with clear diagrammatical representation the formation of the anodic film on an aluminium surface, including the effect of the presence in the aluminium of alloying elements. The caution required in the treatment of castings—both pressure die castings and also sand castings—is emphasised in the foreword.

The first chapter—"Procedure common to all Processes"—will repay most careful consideration by all intending anodisers, for here are minute details of those little points which are so often overlooked (or deliberately ignored) in practice. It has always surprised me that people set out to anodise a series of articles under what they believe to be correct conditions, but, at the same time, by their neglect of detail, they deny themselves the opportunity of producing a good anodic film. For example, two or more separate articles (individually having a good anode connection) may be allowed to make a poor contact one with another, resulting in the formation of a loose oxide film, which in turn inhibits anodising. Again, overcrowding in the anodising tank may cause local overheating, which will result in a poor anodic film being produced. This question of initial care has been stressed in this review because it is so important, and because the matter has been covered so adequately in the A.D.A. book. We, as dyers, are particularly keen that the film, which is intended to be dyed, shall be a good one. We just cannot dye satisfactorily a poor anodic film.

After having read this first chapter, the writer looked with optimism at those to follow, and was not disappointed.

The main anodising processes by chromic, sulphuric, and oxalic acids are detailed and compared.

The dyeing processes are given in good detail, with elaborations of the necessary precautions to be taken, and some suggestions are made concerning suitable dyes to use (and, here, our own Society's *Colour Index* number is quoted where applicable). Besides straightforward dyeing, methods of obtaining many special effects are described, including printing and mottled effects.

The appendixes contain an informative list of patents relating to anodising processes, and the very length of this list should serve as a caution to the intending anodiser. Anodising is fraught with patent difficulties, and much of what is regarded as everyday practice is in fact covered by a patent.

As a critic, and especially one who has found so much to praise, I have searched in these two Bulletins for something to decry—all I asked for was one little error which I might jubilantly expose! Technically, I could find nothing, but, then, I scarcely expected the Association to slip up. I make one suggestion, however, which is that the warning

given on p. 45 of Bulletin No. 14, concerning the possibility of the various tests in the appendixes being superseded by recommendations of the British Standards Institution, should be made clearly to apply to all the tests and not only to the porosity test. The B.S.I. is constantly reviewing its standard testing methods.

Finally, I must pay tribute to the colour plates and other illustrations in Bulletin No. 14.

I do recommend these two books to all those who are new to anodising, and I feel sure that even those who are experienced in the subject will also enjoy reading them.

J. MARSHALL HAY

### The Measurement of Particle Size in Very Fine Powders

By H. E. Rose. Pp. 127. London: Constable & Co. Ltd., 1953. Price, 9s. 6d.

This book contains the material of four lectures delivered at King's College, London. It is unfortunate that it has not been slightly rearranged, as much of the book reads like the transcript of a shorthand record of the spoken lectures. The frequent addition of titles to the names of authors referred to in the lectures is unnecessary, and irritating in a written work. Two minor irritations are the occasional use of undefined symbols in equations, and the mixing of units, such as occurs when particle sizes in millimetres are compared with wavelengths of light in microns, or particle size is quoted in inches and illustrated with a photomicrograph bearing a micron scale.

The first lecture is devoted to a description of the uses of size analysis in various industries, and a discussion of methods of presentation. Some of the statements made are misleading, as, for example, that a reduction of particle size in certain drug suspensions allows more rapid re-suspension in the medium after storage. This is true only when the size reduction is accompanied by flocculation and consequent increase in sediment volume. It is, however, a useful survey.

The second lecture deals at some length with the theoretical basis of sedimentation and elutriation analysis. The practical difficulties of using such methods are not dealt with very adequately, and no guidance is given on the all-important preliminary step of preparing a good stable dispersion of the powder.

The third lecture is mainly concerned with the author's own work on the photo-extinction method of size analysis. The use of an apparatus in which the light-sensitive element subtends a very small solid angle at the suspension, and correction of the observed densities with a factor dependent on particle size, seem to have eliminated or reduced many of the errors inherent in the earlier systems. The author is in error, however, in assuming that the fundamental equations on p. 67 are independent of concentration. The reduction of light intensity in an element  $\delta l$  of suspension is not continuous, but is, in effect, "quantised" in the plane normal to the light beam. Integration up to finite lengths of light beam is, therefore, valid only as the concentration approaches zero. Fortunately, this condition is generally fulfilled for other practical

reasons. The application of the correcting factor, which is highly variable for particle sizes below  $5\mu$ , seems very risky at the lower end of the size range, but for suspensions containing little very fine material the method is worthy of careful study. The remainder of lecture three concerns permeability methods.

Gas adsorption and miscellaneous methods of limited application are discussed in lecture four, together with the microscope. The latter is hardly

given fair prominence in this work, considering the large amount of size analysis which is carried out in industry and research by its use. The shapes of particles used to prove that microscopic counting can give rise to biased errors would certainly give rise to equally large errors in any other method of analysis, and the microscope has at least the advantage of letting the operator know that unusually shaped particles are present.

A. C. COOPER

## Abstracts from British and Foreign Journals and Patents

*The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

### I—PLANT; MACHINERY; BUILDINGS

#### PATENTS

#### Solvent Degreasing of Wool. Smith, Drum & Co.

Machine for degreasing raw wool so as to leave a controlled amount of grease in the wool. *USP 2,621,506*  
C. O. C.

#### Apparatus for the Wet Spinning of Polyacrylonitrile Fibres. American Cyanamid Co.

Continuous Processing of Thread. Courtaulds.  
*BP 695,526*

The thread is formed into a helix on a thread-advancing reel. In the supporting surface of the reel there are longitudinal openings, through which a liquid flows into contact with the thread. This liquid forms a thin-walled film which flows through, in, and around the thread helix, permeating and scouring it to yield a yarn of improved physical characteristics.  
C. O. C.

#### Treating Travelling Filaments with Fluids. DuP.

An improved spinning cell exit door and aspiration take-off device for use in the dry or the evaporative spinning of filaments from spinning solutions.  
*BP 695,571*  
C. O. C.

#### Thread Singeing. J. V. Royes.

The thread passes through an open groove lined at its bottom and sides with an electrical heating resistance. This results in uniform application of the heat around the thread.  
*BP 696,395*  
C. O. C.

#### Wet Treatment of Fabrics. P. F. van Vlissingen & Co.'s Katoenfabrieken.

In a machine in which cloth is led through liquor by means of a set of guide rollers in the liquor and a set of driving rollers above it, each driving roller has two conical surfaces directed towards one another so that the smallest diameter of the roller is in the middle. All the driving rollers are identical. The cloth is fed on to one of the conical surfaces of the first driving roller, and thence via the appropriate guide rollers successively to the other guiding rollers. As it shrinks during treatment it tends to migrate to the centres of the driving rollers to maintain even tension in it. By adjusting the rate of feed relatively to the speed of the driving rollers, the cloth can be passed through the liquor in absence of tension. Alternatively, any desired tension can be imparted to the cloth by passing it through parallel guide rods which force it on to a particular part of the conical surface of each driving roller.  
*BP 694,158*  
C. O. C.

#### Mercerising Machine. E. P. Mullen.

The caustic lye and/or the washing water are applied to the upper surface of the cloth while it is passing through a tenter. The lye is sucked through the cloth and reused. This enables mercerisation to be carried out without altering the length or width of the cloth.  
*USP 2,620,544*  
C. O. C.

#### Piece Scouring. F. Köstring.

The fabric is passed at open width through a vat in which the liquor is violently agitated by a number of vertically reciprocated plungers, and then through a rinsing vat in which the water is similarly agitated. The fabric enters the machine faster than it leaves, so that it passes through without tension.  
*BP 693,207*  
C. O. C.

#### Jig Dyeing. Celanese Corp. of America.

Uniformity and levelness of dyeing are improved by tilting the fabric, as it leaves the liquor, and/or the roll into which the fabric is being wound.  
*BP 695,349*  
C. O. C.

#### Dye Winch. Mellor Bromley & Co.

The liquor is withdrawn from the beck, passed through a tunnel or chamber housing a propeller, and then delivered to the opposite side of the beck at a higher level than that at which it is withdrawn. Circulating the liquor in this manner gives more uniform dyeing, enables the dye liquor to be kept at a uniform temperature throughout the beck, reduces the time needed for dyeing, and makes possible the use of a short liquor ratio, thus enabling the winch to be rotated more slowly and so reducing the tension in the fabric being dyed.  
*BP 693,106*  
C. O. C.

#### Expander and Contractor Rolls. J. D. Robertson.

A series of roll sections have cylindrical bearings on cylindrical journals which are non-rotatably mounted on a curved shaft. The roll sections and the journals are kept from moving along the shaft by means placed at the ends of the shaft.  
*BP 695,163*  
C. O. C.

#### Hydraulic Drive for Decatising Machines or the like.

David Gessner Co.  
*USP 2,618,954*  
Separate hydraulic driving motors are provided for the perforated drum and the storage roll, each motor when inactive acting as a brake on the associated idle drum or roll.  
C. O. C.

#### Measuring Moisture in Material. DuP.

Several pairs of electrodes are applied to the material at regulated distances between each pair, the variation in the resistance offered by the material to a current passed between the two electrodes of each pair being automatically recorded. Examples of apparatus suitable for application to warps or paper are described.  
*USP 2,621,232-3*  
C. O. C.

#### Measuring or Controlling Humidity. P.A.M.

A member capable of extension according to humidity, e.g. a piece of parchment or vellum, is mounted on a frame made up of two rods connected by a bridge member at one end and a plate at their other ends. The frame is made of materials having compensating coefficients of expansion, so that the efficiency of the apparatus is not affected by changes in temperature. The member sensitive to humidity actuates a switch which works an indicator and/or means controlling the humidity.  
*BP 695,579*  
C. O. C.

#### Chambers for the Drying or Gaseous Treatment of Textiles. Gebrüder Sucker.

The chamber, if desired double-walled, has an arrangement for drawing a length of textile through it. The chamber channels contain rotary apparatus for directly sucking off the hot air or gas from outflow channels near the jets which supply the hot air or gas, and guiding it in closed circulation over heaters to the jets. With the same-sized chamber and with use of less energy, higher efficiency and more uniform drying or other treatment is obtained.  
*BP 695,582*  
C. O. C.

**Calender Rolls.** Farrel-Birmingham Co. BP 695,312

The rolls are shaped so that under actual working conditions of temperature and pressure the distance between a pair of co-operating rolls is constant throughout the width of the material being treated. C. O. C.

**Counterbalancing Printing Screens, Trays, and other Objects and Transporting them along Work Tables.** Stead, McAlpin & Co. BP 694,974

The screen or other object is counterweighted at one side so that it is kept in, or biased towards, a raised position above the table. It can be moved on to and away from the table against the action of the counterweight and is movable along a guide extending the length of the table. C. O. C.

**Roller Printing Machines.** Schlieper & Baum.

BP 695,041

A machine in which the effective width of the transfer rollers can be increased continuously to conform with the width of the material being treated without rendering necessary either exchange of the printing rollers or change in width of the pressure cylinder surface. C. O. C.

**Electronic Register Regulators for Multicolour Printing.** Goebel. BP 694,146**Felt-shearing Machine.** Établissements Bruyère.

BP 696,042

The felt is carried by a roller against a cutter having reciprocating blades, the drive for the blades being such that they wear uniformly, thus eliminating the need for frequent grinding. C. O. C.

**Setting Nylon Hose.** L. Heldmaier. BP 694,821

Nylon hose are mounted on forms carried by an endless conveyor through water sprays followed by a preheating chamber, and then a plasticising chamber, in which radiant heat is applied. Preheating the hose and forms enables the setting time to be reduced. G. E. K.

**Mechanical Production of Acoustic Vibrations for Use in Emulsification, Dispersion, etc.** Ultrasonics. BP 695,491**Drying Sheets of Crêpe or other Forms of Rubber.** R. H. Thomas. BP 695,791**Scanning Mechanism for Colour-sorting Machines.** E. H. Bickley. BP 696,246

Treating Flexible Materials with Powder (X p. 391).

**II—WATER AND EFFLUENTS****PATENT****Base-exchange Water-softening Plant.** Filtrators. BP 695,623

Plant specially suitable for treating river or other water which contains small amounts of ammonia. C. O. C.

**III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS****Nomogram for Ammonium Sulphate Solutions.** M. Dixon. *Biochem. J.*, 54, 457-458 (June 1953).

A nomogram for ammonium sulphate solutions is described, from which the amount of solid ammonium sulphate to be added to a solution of known concentration to produce a given degree of saturation may be calculated. This has been found to be of use in the separation of proteins by fractional precipitation. P. G. M.

**PATENTS****Detergent Compositions.** British Oxygen Co. BP 696,031-2

The condensate of 8-16 mol. of ethylene oxide with 1 mol. of a phenol, cresol, or xyleneol, nuclearly substituted by at least one alkyl group of > 7 C, is used together with a disodium dihydrogen pyrophosphate and/or tetrasodium pyrophosphate. If it is desired to convert the product into a dry powder, then it is mixed with 60-300% (on the weight of the synthetic detergent) of a water-insoluble inert, white, inorganic filler, e.g.  $MgCO_3$ . C. O. C.

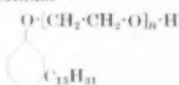
**Neutral Inorganic Esters of Polyglycol Ethers—Auxiliary Agents.** Basf. BP 695,314

The condensates obtained by heating approx. 2 mol. of a compound of formula  $A-(O-C_2H_4)_n-B$  (A = aliphatic, cycloaliphatic, aromatic, or heterocyclic radical of 3-23 C; B = OH or OM (M = alkali metal); n > 2) with 1 mol. of a dibasic inorganic acid containing no C atom, a halide

thereof, or (when B = O-SO<sub>3</sub>H or O-SO<sub>3</sub>M, and M = metal) an alkali-metal sulphide have good wetting and dispersing powers. C. O. C.

**Polyalkylene Glycol Ethers—Auxiliary Agents.** Ciba. BP 695,036

Compounds of formula—



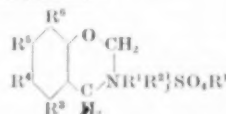
are used as washing, emulsifying, levelling, or wetting agents, etc. Those obtained by condensing a monohydroxy six-membered homocyclic compound with 12-20 mol. of ethylene oxide will remove mineral oil from wool in presence of acid. C. O. C.

**Detergent Mixtures containing β-Aminopropionates.** General Mills. USP 2,619,467

Addition of a hydrophilic surface-active agent to a mixture of detergents, at least one of which is of formula  $R-NH-CH_2-CH_2-COOB$  (R = aliphatic hydrocarbon of 12-18 C; B = water-solubilising cation), much improves the surface-active and detergent properties of the mixture. C. O. C.

**Quaternary Ammonium Salts having Surface-active Properties.** ICI. BP 695,550

Compounds of formula—



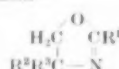
(R<sup>1</sup> = CH<sub>3</sub> or C<sub>2</sub>H<sub>5</sub>; R<sup>2</sup> = sat. aliphatic hydrocarbon radical which may be subst. by OH; R<sup>3</sup> = Hal or a sat. aliphatic radical; R<sup>4</sup>, R<sup>5</sup>, and R<sup>6</sup> = H, Hal, or sat. aliphatic hydrocarbon radicals) are useful as cationic surface-active agents, textile treatment agents, disinfectants, and moth-proofing agents. C. O. C.

**Plasticisers, Emulsifiers, and Textile Auxiliary Agents.** Henkel & Cie. BP 694,633

The products obtained by the simultaneous action of Cl<sub>2</sub> and SO<sub>2</sub> on sat. aliphatic or cycloaliphatic hydrocarbons (which may or may not be substituted by Hal or Ar) are treated below 0°C. with aqueous ammonia containing at least 30% by wt. of NH<sub>3</sub>. The resulting sulphonamides and/or disulphonamides are used as textile auxiliary and emulsifying agents and as plasticisers. C. O. C.

**Conditioning Cellulose Acetate Yarn.** Eastman Kodak Co. USP 2,622,045

Compounds of formula—



(R<sup>1</sup> = Alk or alkenyl of 10-20 C; R<sup>2</sup> = CH<sub>3</sub> or C<sub>2</sub>H<sub>5</sub>; R<sup>3</sup> = H or CH<sub>3</sub>) when added to yarn lubricants greatly improve their antistatic and lubricating properties. C. O. C.

**Compositions for Hair Waving.** Gillette Safety Razor Co. BP 695,797

Sodium perborate monohydrate A (exact constitution unknown, but made by mixing boric acid or an alkali-metal borate with H<sub>2</sub>O<sub>2</sub>, an alkali-metal peroxide or hydroxide, and water to a homogeneous solution and then evaporating to dryness) in dilute aqueous solution is an excellent agent for use in the neutralisation step. C. O. C.

**Polymeric Hydroxyl-containing Titanium Carboxylates—Dispersing Agents for Pigments and Water-repellent Agents.** DuPont. USP 2,621,194

The products obtained by interaction of a titanium tetrahalide, a long-chain aliphatic monocarboxylic acid of 8-20 C, and water are very soluble in organic solvents, and such solutions act as dispersants for carbon black and other inorganic and organic pigments. They are also useful for imparting a water-repellent finish to fabrics. C. O. C.

**Stable Artificial Resin Emulsions.** Ciba. BP 694,974

Stable emulsions of resins suitable as textile finishes and for fixing pigments on textiles are obtained by emulsifying in water, in presence of a reaction product of a protein with

an alkylene oxide, an organic-solvent solution of a hydroxymethyl compound of urea or the like in which the O atom of at least one hydroxymethyl group is bound to an organic residue of  $> 3$  C.

C. O. C.

**Mothproofing.** E. B. Eriksson. *BP* 695,168

Better fixation of silicofluorides to wool is obtained if the wool is treated with alkali to neutralise any acid present and then well rinsed in water before application of the silicofluoride. The silicofluoride is so firmly bound to the wool that after the first wash the fluorine content does not diminish on subsequent washings.

C. O. C.

**Whitening and Brightening Rinse for Nylon.** Pro-Nyl Chemicals. *USP* 2,619,470

A concentrated rinse composition for nylon consists of "3:7-dianisoylaminodibenzothiothiophenylsulphonic acid" or its sodium salt dispersed in water containing  $< 20\%$  of a simple monohydric alcohol.

C. O. C.

**Wrinkle Coating Compositions.** New Wrinkle.

*BP* 695,581

A coating composition made up of vinyl chloride-vinyl acetate copolymer (150 parts by wt.), butyl acetate (100), ethyl methyl ketone (100), and cyclohexanone (125) is very suitable for application by the process of *BP* 692,888 (J.S.D.C., 69, 356 (Sept. 1953)).

C. O. C.

**Stabilised Sodium Hydrosulphite.** YDC. *BP* 695,375

Atmospheric deterioration of sodium hydrosulphite is reduced by intimately mixing with it 0.1-2.0% of a liquid ester of low volatility derived from alcohols, containing  $> 4$  C. Thus the purity of a sample of sodium hydrosulphite treated with 1% dimonyl phthalate, after standing for 7 days in an open container, fell from 94.5% to 81.0%,  $\text{Na}_2\text{S}_2\text{O}_4$ , whereas that of a simultaneously exposed untreated sample fell to 64.4%  $\text{Na}_2\text{S}_2\text{O}_4$ .

R. K. F.

**Carrotting Solution.** Frank H. Lee Co. *BP* 695,135

An aqueous solution containing, by weight, 3-5% of a non-hydrolysing oxidising agent, 1.0-4.5% of a hydrolysing acid, and 0.1-0.4% of tungstophosphoric acid renders fur feltable without impairing the strength or wearing properties of the final product.

C. O. C.

**Solvents and Plasticisers for Polyacrylonitrile Polymers and Interpolymers.** Röhm & Haas.

*BP* 694,827

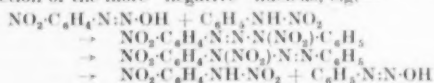
Aminoacetonitrile and its *N*-substituted derivatives are good solvents and plasticisers for polyacrylonitrile polymers and interpolymers.

C. O. C.

#### IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

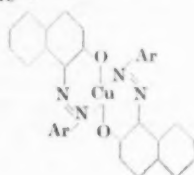
**1:3-Diaryl-1-nitrotriazens.** N. M. Baranchik, I. V. Grachev, and D. Z. Zavel'sky. *Doklady Akad. Nauk S.S.S.R.*, 89, 1007-1009 (21 April 1953).

In the reaction in weakly acid soln. of phenylhydrazine with 4-nitro-, 2-chloro-4-nitro-, or 4-chloro-2-nitro-benzenediazonium chloride, the corresponding nitro- or chloronitro-phenylhydrazine is isolated and the formation of benzenediazonium chloride is demonstrated. It is considered that the 1-nitrotriazene first formed isomerises to the 3-nitrotriazene by migration of the nitro group in the direction of the more "negative" nucleus, e.g.—



A. E. S.

**Copper Complexes of Substituted 1-Phenylazo-2-naphthols.** I. Kadugai. *Bull. Research Council Israel*, 1, (4), 96 (1952); *Chem. Abs.*, 47, 6813 (25 July 1953). *o*-Hydroxyazo dyes form copper compounds having the following structure—



Ability of the N atoms to participate in the complex formation is influenced by substituents in the Ar nucleus. Thus equivalent amounts of 1-phenylazo-2-naphthol and tetra-amminocupric sulphate form the Cu complex at 200°C. ( $\text{NH}_3$  evolved); 1-*p*-nitrophenylazo-2-naphthol does the same at 100°C.; and 1-*o*-dinitrophenylazo-2-naphthol at 60°C. The Cu compounds are obtained practically pure by treatment with water, alcohol, ether, acetone, and pyridine; they are dark brown insoluble powders.

C. O. C.

**New Method of Emission of Light by some Organic Compounds.** A. Bernanose, M. Comte, and P. Vouaux. *J. Chem. phys.*, 50, 64-68 (1953); *Chem. Abs.*, 47, 6258 (10 July 1953).

Gonarine and Acridine Brilliant Orange E, when adsorbed on Cellophane film which is placed in a sufficiently strong alternating electric field, give rise to strong light emission. The phenomenon has a voltage threshold of 400-800 v., and the intensity of the light emitted increases as the voltage rises to 2500 v. The electric field causes formation of metastable excited molecules, which emit light as they return to their ground state.

C. O. C.

**Dyes for Cellulose Acetate. III—1-Alkylamino-2-alkoxyanthraquinones.** K. Naiki and S. Tsuruoka. *J. Soc. Org. Synthetic Chem. (Japan)*, 11, 113-117 (1953); *Chem. Abs.*, 47, 7217 (25 July 1953).

Preparation of the *NN*-dimethyl, *N*-ethyl, and *N*-methyl derivatives of 1-amino-2-methoxyanthraquinone is described. Their affinity for and fastness on cellulose acetate follow the above order. The corresponding 2-ethoxy and 2-butoxy compounds behave similarly.

C. O. C.

**Synthesis of Heterocyclic Nitrogen Compounds.**

**LXIV—Photosensitising Dyes—4.** T. Takahashi, H. Yamagihara, K. Satake, K. Mori, and S. Boku. *J. Pharm. Soc. Japan*, 72, 291-296 (1952); **LXX—Photosensitising Dyes—5.** T. Takahashi and K. Satake. *Ibid.*, 463-468. **LXXI—Photosensitising Dyes—6.** *Ibid.*, 468-470; *Chem. Abs.*, 47, 6401 (10 July 1953).

**Effect of Various Substances on the Rate of Photo-oxidation of Chlorophyll a.** V. B. Evstigneiev and V. A. Gavrilova. *Doklady Akad. Nauk S.S.S.R.*, 89, 523-526 (21 March 1953).

The fading of chlorophyll a when irradiated (filament lamp; filter "RG-2") in toluene soln. is appreciable only in presence of air. The presence also of small amounts of water, alcohol, aniline, or quinoline greatly accelerates fading; ether and acetone are without effect; and pyridine, piperidine, nicotine, and hexamethylenetetramine greatly reduce the rate of fading. There is no parallel between these effects and the immediate effects produced by these substances on the absorption and fluorescence spectra of chlorophyll.

A. E. S.

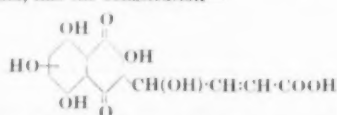
**Melanin. I—The Melanin of Sepia.** L. Panizzi and R. Nicolais. *Atti. Accad. nazl. Lincei, Rend. Classe sci. fis., mat. e nat.*, 12, 420-427; *Gazz. Chim. ital.*, 82, 435-460 (1952); *Chem. Abs.*, 47, 6868 (25 July 1953).

An account of the reaction of melanin from the sepia of the squid. Unlike melanin prepared by oxidising tyrosine, it yields a small amount of a pyrroletricarboxylic acid when treated with  $\text{KMnO}_4$  in alkaline solution.

C. O. C.

**Derivatives of Naphthaquinone. X—The Pigments from the Sea Urchins—5.** C. Kuroda and M. Okajima. *Proc. Japan Acad.*, 27, 343-345 (1951); *Chem. Abs.*, 47, 6386 (10 July 1953).

Evidence is given indicating that Spinochrome  $\text{M}_1$ , the pigment from the spines of the sea urchin, *Anthocidaris crassispina*, has the constitution—



C. O. C.

**Precipitation of Light-coloured Cadmium Yellow in Acid Medium.** E. Stock and K. Ledermann. *Deut. Farben-Z.*, 7, 87-90 (1953); *Chem. Abs.*, 47, 6669 (10 July 1953).

A detailed discussion of the composition, manufacture, fastness to light, hiding power, oil absorption, drying

properties, compatibility with lead pigments, and chemical resistance of cadmium pigments. In the direct method of precipitation CdS is pptd. at once by  $H_2S$  from conc.  $CdCl_2$  or  $CdSO_4$  soln. but more slowly from dil. soln. The first ppt. is light yellow. Poor agitation produces a ring of dark ppt. at the meniscus. The ppt. tends to deepen in colour and may even turn orange. Further deepening occurs on washing with hot water and on drying above  $90^\circ C$ . Acid added before pptn. tends to produce a deeper colour, HCl being more active than  $H_2SO_4$ . No light-coloured ppt. result from conc. soln. of Cd salts, but deepening of colour occurs also in dil. soln. No pale colours result from hot soln. Protective colloids have no effect. Light-coloured Cd yellow can be made by direct pptn. from acid soln., the most favourable conditions being—conc. 8% of  $3 CdSO_4 \cdot 8 H_2O$  (wt./total vol.), acidity 4–8% additional  $H_2SO_4$ , pptn. temp.  $15^\circ C$ , wash water temp.  $75^\circ C$ , maximum drying temp.  $60^\circ C$ . To remove the colour the ppt. should be filtered by suction. Even without additional  $H_2SO_4$ ,  $CdSO_4$  yields light-coloured CdS, but  $CdCl_2$  cannot replace  $CdSO_4$ . Yellow CdS was analysed and found to consist of 76.28% Cd, 21.26% S, 1.60%  $SO_4$ , and 0.85%  $H_2O$ . Dark CdS (from HCl soln.) contained 76.86% Cd, 21.17% S, 1.22% Cl, and 0.75%  $H_2O$ . The hue of these pigments ranged from 2 pa to 4 pa of the Ostwald table. The dark pigment had greater hiding power than the light pigment. Let-downs with blanch fix indicated that 50–60% of it may be added without impairing hiding power. C. O. C.

**Inorganic Pigments. III—Cobalt Green—Relation between  $CoO$  and  $Co_2O_3$  and the Reaction between Cobalt Oxides and Zinc Oxide.** T. Ando and R. Umenoto. *Bull. Osaka Ind. Research Inst.*, 2, 89–93 (1951); *Ceram. Abstr.*, 151–152 (1952) (in *J. Amer. Ceram. Soc.*, 35, (8), 6669 (10 July 1953)).

Decomposition of  $Co_2O_3$  to  $CoO$  occurs rapidly at  $940^\circ C$  in air, but at  $700$ – $850^\circ C$ , no decompositions occurs.  $CoO$  obtained by decomposition of  $Co_2O_3$  at  $1100^\circ C$  does not oxidise appreciably when heated in air at  $500$ – $800^\circ C$ ;  $CoO$  obtained by heating  $CoCO_3$  in  $CO_2$  at  $600^\circ C$  oxidises rapidly in air at  $600^\circ C$ .  $CoO$ – $ZnO$  solid soln. are formed with difficulty on heating mixtures of  $CoO$  from  $Co_2O_3$  and  $ZnO$ , but readily on heating  $CoCO_3$  and  $ZnO$  in  $CO_2$ . The X-ray powder diagrams of the two forms of  $CoO$  are identical. C. O. C.

**Crestmore Sky-blue Marble, its Linear Thermal Expansion and Colour.** J. L. Rosenholtz and D. T. Smith. *Amer. Mineralogist*, 35, 1049–1054 (1950); *Chem. Abs.*, 47, 6826 (25 July 1953).

The blue of marble from Commercial Quarry, Crestmore, California, starts to fade when heated to  $225^\circ C$ , and changes to a light cream at  $275^\circ C$ , suggesting that the blue is due to residual strain. C. O. C.

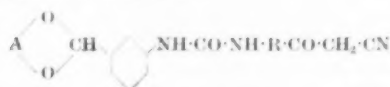
## PATENTS

**Introduction of Alkyl Groups into Cyclic Compounds.**

Basf. *BP* 694,072  
Alkyl groups are introduced into cyclic compounds by treating the latter with aliphatic alcohols or alkali-metal alkoxides in molten  $AlCl_3$ –alkali-metal chloride. Thus, all parts being by weight, methanol (8) is added over 1 hr. to isodibenzanthrone (25) in a melt of  $AlCl_3$  (200) and  $NaCl$  (30) at  $130$ – $135^\circ C$ . When no further change is noted in a sample, the product, a violet vat dye containing ca. 4  $CH_3$  groups, is isolated by pouring into water. R. K. F.

**Acetals of Cyanoacetylarylureidobenzaldehydes.**

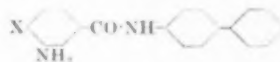
DuP. *BP* 695,164  
Compounds of formula—



(R = bivalent aromatic cyclic radical, A = carbon chain of a monomeric or polymeric polyhydric alcohol having the OH groups which form the acetal on C atoms which are either adjacent or separated by one C atom) are exceptionally good magenta colour formers. C. O. C.

**Monoazo Pigments.** O. F. Schulz and P. H. Fickel. *BP* 695,334

Diazo compounds of amines—



(X = Alk, O-Alk, or Hal) coupled with arylides of 2-hydroxycarbazole-3-carboxylic acid, of 2-carboxy-3-hydroxydiphenylene oxide, or of 2-carboxy-3-hydroxydiphenylene sulphide, the components being so selected that they contain no water-solubilising group, yield brown, brown-violet, and blue-grey pigments, similar to the red and violet pigments of *BP* 679,275 (cf. *J.S.D.C.*, 68, 525 (1952)), in which the same diazo compounds are coupled with arylides of 3-hydroxy-2-naphthoic acid. Thus *p*-(3-amino-4-methoxybenzamido)diphenyl is diazotised and coupled with the *p*-chloroanilide of 2-hydroxycarbazole-3-carboxylic acid. The brown pigment so produced colours polyvinyl chloride with good fastness to bleeding. E. S.

**Bright Red Disazo Acid Dyes from Diaminodiphenyl Sulphides and Sulphoxides.** Gy. *BP* 695,768

2:2'-Diamino-4:4'-dihalogenodiphenyl sulphides or sulfoxides are tetrazotised and coupled with 2 mol. of 2-amino-8-naphthol-6-sulphonic acid under acid conditions, so that coupling takes place in the 1-position to produce bright red acid dyes. Thus 2:2'-diamino-4:4'-dichlorodiphenyl sulphide is treated with nitrosylsulphuric acid, and the tetrazo solution, after dilution with ice and partial neutralisation, is coupled with a cold acetic-acid solution of 2-amino-8-naphthol-6-sulphonic acid in presence of sodium acetate. The diazo dye so formed dyes wool bluish red from a neutral or weakly acid bath. E. S.

**Bright Blue and Blue Green Disazo Dyes.** B. Gaspar and P. D. Dreyfuss. *USP* 2,612,496

In bright blue and blue-green dyes of general formula—  
Acyl-NH-A<sup>1</sup>(OH)-N=N-B-N=N-A<sup>2</sup>(OH)-NH-Acyl

(A<sup>1</sup> and A<sup>2</sup> = naphthalene nuclei in which the OH is *ortho* to the azo link, and which may contain  $SO_3H$ ; B = aromatic nucleus in which the azo links are *para* to each other, and which preferably also contains two alkoxy groups *para* to each other) the fastness to light depends largely on the acyl group. Thus the disazo compound 1:4-diamino-2:5-dimethoxybenzene-2(2:4-dichlorobenzoyl-H acid)<sub>2</sub> is faster to light than the corresponding dye from toluenesulphonyl-H acid. Such dyes neither diffuse nor desensitise a photographic emulsion, and they give colourless reduction products in dye-bleaching processes of colour photography. They also dye vegetable and animal fibres. E. S.

**Navy Blue and Black Copperable Trisazo Direct Cotton Dyes.** Ciba. *BP* 695,330

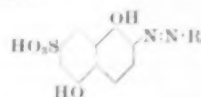
Trisazo dyes for coppering on cellulose fibres are made by diazotising an aminodisazo compound—



(R<sup>1</sup> = a heterocyclic residue containing a five-membered ring containing at least two hetero atoms of which at least one is N; R<sup>2</sup> = a residue of the benzene or naphthalene series, preferably containing, *ortho* to the  $NH_2$ , a group (e.g. alkoxy) capable of taking part in metal-complex formation; R<sup>3</sup> = a naphthalene residue in which the  $NH_2$  is in the 1 or 2 position (attached directly or through a bridge member), an OH group being in the 5-position, and an  $SO_3H$  in the 7-position) and coupling with a component capable of metal-complex formation (e.g. salicylic acid and especially 8-hydroxyquinoline). Thus the trisazo dye 6-aminoindazole- $\rightarrow$ *p*-cresidine- $\rightarrow$ 2-(4'-aminophenyl)-amino-5-naphthol-7:3'-disulphonic acid- $\rightarrow$ 8-hydroxyquinoline gives navy blues on cotton by the single-bath or two-bath after-coppering process. E. S.

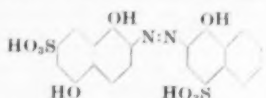
**Grey Copperable Trisazo Direct Cotton Dyes.** S.

*BP* 695,534  
Grey direct dyes are made by coupling tetrazotised *o*-diamidine with 1 mol. of the copper complex of a monazo compound—



(R = residue of a naphthol, of a naphthol-mono-, -di-, or -tri-sulphonic acid, or of a sulphonated or unsulphonated pyrazolone which has been coupled *ortho* to an OH group)

and with 1 mol. of the same or any other coupling component. The triazo dyes so formed may be coppered in substance or on the fibre. Thus, tetrazotised *o*-dianisidine is coupled first with an alkaline solution of 1 mol. of 1-naphthol-3:6-disulphonic acid, and secondly with 1 mol. of the copper complex of the monazo compound—



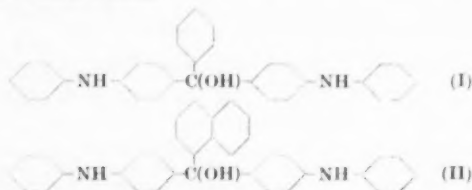
in presence of ammonia and pyridine. The triazo compound so formed is isolated and demethylated by a coppering treatment. The copper complex so formed dyes cotton blue-grey.

E. S.

**Triphenylmethane Dyes.** FBy.

BP 694,681

Dyes derived from compounds of formula I or II (in which each of the two benzene rings remote from the central C atom is substituted by one or more acid groups or by Alk. subst. by an acid group), when used in photographic antihalation and filter layers, bleach out in alkaline developing baths but resist alkali when treated with weakly alkaline emulsion layers. Where the dye is derived from I at least one of the benzene rings linked to the central C atom is substituted by Alk or Hal in the *ortho* position to the central C atom.



C. O. C.

**Bis(halogenophenoxyphenylamino)-anthraquinones—Acid Dyes.** S.

BP 694,713

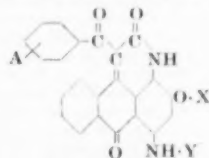
*aa*-Di-halogeno (or nitro, amino, or hydroxy)anthraquinones, possibly containing one or two additional Hal or OH groups, are condensed with 2 mol. of 4-amino-4'-halogeno-diphenyl ether and sulphonated to produce violet to green dyes for wool, silk, and nylon, having good neutral-dyeing properties. Thus a mixture of leuco-1:4-diaminoanthraquinone and 1:4-dihydroxyanthraquinone is heated at 95–105°C. for 15–16 hr. with 4-amino-4'-chlorodiphenyl ether and butyl alcohol in the presence of HCl and H<sub>3</sub>BO<sub>3</sub>. After making alkaline with NaOH and filtering, the product is sulphonated in 100% H<sub>2</sub>SO<sub>4</sub> at 20–40°C.

R. K. F.

**Anthrapyridone (3-Azabenzanthrone) Acid Dyes.** S.

BP 694,668

Sulphonated derivatives of the dye base—



(A = H, CH<sub>3</sub>, Cl, Br, OCH<sub>3</sub>, or NO<sub>2</sub>; X = C<sub>6</sub>H<sub>5</sub>, Alk-C<sub>6</sub>H<sub>4</sub>, diphenyl, or naphthyl-phenyl; Y = C<sub>6</sub>H<sub>5</sub> or C<sub>10</sub>H<sub>7</sub>) are red acid dyes. The base is made, e.g. by heating 1-amino-4-anilino-2-*p*-tert.-amylphenoxyanthraquinone and ethyl benzoylacetate in nitrobenzene containing Na<sub>2</sub>CO<sub>3</sub> at 130°C. for 24 hr., and separating by cooling and adding isopropyl alcohol.

R. K. F.

**Carbazole Vat Dye.** Ciba.

BP 693,075

The tetra-anthrime—



is heated at 120–140°C. for 45 min. in a tertiary base, e.g. pyridine, containing AlCl<sub>3</sub> to give the corresponding grey carbazole dye.

R. K. F.

**Symmetrical Thioindigoid Dyes.** Ciba.

BP 692,962

2:5-Dimethyl- or 2-chloro-3:5-dimethyl-phenylthioglycolic acid is chlorinated in a solvent inert to chlorosulphonic acid and, by adding the latter without isolating the intermediate product, is converted to the corresponding hydroxythionaphthen or thioindigoid dye according to the temperature used. Thus 2:5-dimethylphenylthioglycolic acid, sulphuryl chloride, and SbCl<sub>5</sub> are heated at 60°C. for 1 hr. in tetrachloroethane. After cooling to 30°C., chlorosulphonic acid is slowly added. The resulting 5:5'-dichloro-4:4':7:7'-tetramethylthioindigo is separated by pouring into ice and water and steam-distilling. Alternatively, the chlorosulphonic acid is added at 0°C. to give 5-chloro-3-hydroxy-4:7-dimethylthionaphthen.

R. K. F.

**Polyazine Dyes.** Calico Printers Assocn.

BP 694,451

An aromatic *m*- or *p*-dialdehyde or a tetra-acetate thereof (oxidation of corresponding dimethyl compound with CrO<sub>3</sub>·H<sub>2</sub>SO<sub>4</sub> in presence of acetic acid-acetic anhydride) is condensed with hydrazine, its hydrate, or a salt thereof to give a yellow or orange water-insoluble pigment suitable for the resin-printing of textiles. Alternatively, the pigment may be formed on the fibre. Thus a cotton fabric is padded with a 5% aqueous-ethanolic solution of terephthalaldehyde at 80°C. It is then, without being dried, passed through 5% aqueous hydrazine sulphate at 90°C., when immediate formation of the colouring matter takes place.

R. K. F.

**Styryl Methin Dyes.** Ciba.

BP 694,754

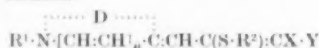
Dyes of formula  $Pz(CH:CH-R)_nX$  (Pz = pyrazolium nucleus; *n* = 1 or 2; R = an aromatic radical attached to position 3 and/or 5 of the pyrazole nucleus, being the same or different when *n* = 2; X = an anion) have excellent photographic sensitising properties and excellent ultraviolet absorption.

C. O. C.

**Dyes useful as Photographic Sensitisers.** Ilford.

BP 694,744

Dyes of formula—



(R<sup>1</sup> and R<sup>2</sup> = Alk or aralkyl; D = atoms to complete a 5- or 6-membered ring; *n* = 0 or 1; X = cyano, acyl, or carbalkoxy; Y = cyano, carbalkoxy, carbamoyl, or N-subst. carbamoyl) are useful as photographic sensitisers.

C. O. C.

**Pyrococline Dyes.** Eastman Kodak Co.

USP 2,622,982

Dyes of formula—



(R<sup>1</sup> and R<sup>2</sup> = H, Alk of 1–4 C, or Ar of the benzene series; R<sup>3</sup> and R<sup>4</sup> = same or different Alk; Z = atoms to complete a pyridine or isoquinoline residue; X = anion; *n* = 0 or 1) are photographic filter and anti-halation dyes which do not bleed and are readily bleached during processing. They are formed by condensing a suitable pyrococline and an aldehyde in presence of an anhydrous acid.

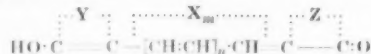
C. O. C.

**Polymethin Dyes having Affinity for Animal Fibres.**

Gevaert.

USP 2,620,339

Dyes of formula—



(*m* and *n* = 1 or 2, *n* being < *m*; Y and Z = atoms to complete a carbocyclic or heterocyclic ring in which one or more groups of two atoms may be shared by an additive ring; X<sub>m</sub> = atoms to complete a carbocyclic or heterocyclic ring, which may have a fused-on arylene group, with any two carbon atoms of the polymethin chain; X, Y, and Z may be the same or different) are substantive to animal fibres.

C. O. C.

**Non-dusting Powders.** YDC.

BP 693,765

The tendency of finely divided powders to form dust clouds is reduced by admixture of 0.1–2.0% of a mixture of a

mineral oil and an emulsifying agent, e.g. triethanolamine oleate.

BP 695,372

The dust associated with dye and other powders is eliminated or reduced by intimately mixing into them 0.1–2.0% of a liquid ester having  $> 5$  C atoms and comprising completely esterified alcohols containing  $> 4$  C, e.g. diethyl phthalate.

R. K. F.

#### Vat Dye Preparations. Basf.

BP 694,473

Vat dye pastes and powders containing the dye in a state of division, which lies below the limit of visibility in an optical microscope, are prepared by precipitating the free leuco compound from a hydrosulphite vat by acidifying in the presence of a dispersing agent, e.g. the condensation product of naphthalene- $\beta$ -sulphonic acid and formaldehyde. After being filtered off, the resulting leuco derivative is kneaded in viscous form with a non-flocculating dispersing agent, e.g. sulphite cellulose liquor. Pastes are prepared by then adding water, while powders result on drying and grinding.

R. K. F.

#### Carbon Black. Shell Refining & Marketing Co.

BP 695,492

A carbonaceous material is burnt with a limited quantity of air, and the products of combustion are cooled by contact with a finely divided inert solid which itself is cooled by a medium which does not come into contact with the product of combustion. The carbon black is then separated from the other products of combustion.

C. O. C.

#### Furnace Black. Columbian Carbon Co.

BP 694,477

An improved process which avoids limiting either the furnace width or the relative volumes and mass velocities of make gas and the blast flame gases.

C. O. C.

#### Reducing Metal Powders. Charles Hardy.

USP 2,621,137

A process of brightening or annealing metal powders by heating in a reducing atmosphere without producing sintering or agglomeration of the particles. It is especially useful for copper, iron, and bronze powders.

C. O. C.

#### Iron Oxide Pigment. C. K. Williams & Co.

USP 2,620,261

Orange and red hydrohematitic iron oxide pigments are produced by keeping at 60°C. a mixture of precipitated brown ferric hydroxide, a solution of a ferrous salt, and an ionisable compound of Zn or Cu, there being at least one mol. of the last-named to 10 mol. of the ferrous salt, until the product has the desired hue. The pigment is then separated and dried without roasting.

C. O. C.

#### Arsenic-activated Zinc Sulphide Phosphors. British Thomson-Houston Co.

BP 696,188

Incorporation of 0.01–2.0% of arsenic in ZnS yields a phosphor having white luminescence in ultraviolet radiation.

C. O. C.

Chemical Identification of Vegetable Dyes used on Ancient Japanese Silk (Preliminary Report) (VIII p. 388).

## V—PAINTS; ENAMELS; INKS

**Adsorption of Dyes from Aqueous Solutions on Pigments.** W. W. Ewing and F. W. J. Liu. *J. Colloid Sci.*, **8**, 204–213 (1953); *Chem. Abs.*, **47**, 6215 (10 July 1953).

Samples of anatase, rutile, and ZnO, whose surface areas were known from electron-microscope or nitrogen-adsorption measurements, were used as adsorbents for purified C.I. 681 and 151 from aq. soln. at room temp. One isotherm was also determined at 60° and 93°C. The results were consistent with the Brunauer theory of multi-layer adsorption. The areas calculated for the adsorbents were consistent with known values of the cross-sectional areas of the adsorbates, viz. 80 sq. a. for C.I. 681 and 70 sq. a. for C.I. 151. The areas calculated from the projection of the models are 160 and 140 sq. a. respectively. It is thought that this discrepancy can be accounted for by formation of a bimolecular layer.

C. O. C.

#### PATENTS

#### Steam-setting Printing Ink. Sun Chemical Corp.

USP 2,621,130

A steam-setting ink which is readily removed from the press distributing mechanism by aqueous wash-up materials contains as vehicle a water-insoluble resinous binder

dissolved or dispersed in a water-miscible liquid, together with a water-dispersible non-ionic surface-active agent.

C. O. C.

#### Ink for producing Colour-variable Writing, etc. D. L. Merritt.

BP 695,548

The ink contains two or more colouring materials in such proportions that its colour is appreciably different from that of any of its constituent colouring materials. The printed or written matter is then treated with a bleaching agent which bleaches at least one of the colouring materials but has no effect on at least one of them.

C. O. C.

#### Phenol Aldehyde Resin Compositions for protecting Ferrous Metals against Corrosion. Brycel.

BP 695,552

A composition comprising a Novolac resin, ferric chloride and/or bromide, a common organic solvent, and a small amount of an alkali combines with the rust on the surface of the metal and simultaneously forms a film giving protection against external attack; e.g. 50 g. o-cresol Novolac resin (acid condensed), 50 g. methanol, 10 g. ferric chloride in 30 g. methanol, 2 g. aq. ammonia (sp. gr. 0.950), and 5 g. tritoyl phosphate yield a brown mixture which on application to rusty iron yields a glossy and very hard dead black film.

C. O. C.

#### Coating Composition. Ideal Chemical Products.

BP 696,087

A composition for spraying or spreading on metal, wood, or concrete contains chlorinated rubber (as vehicle), a highly chlorinated paraffin wax, a fire-resistant plasticiser, a fire-resistant adhesive, a pigment, and a volatile aromatic solvent. It is resistant to fire and corrosion and is highly adhesive.

C. O. C.

Wrinkle Coating Compositions (III p. 384).

Fast Diazo Prints (IX p. 390).

Dyes for Photographic Filters and the Discharge of such Dyes (IX p. 390).

## VI—FIBRES; YARNS; FABRICS

**Behaviour of Keratin Fibres in Thioglycollate Systems.** H. Freytag. *Z. Naturforschung*, **76**, 645–655 (1952); *Chem. Abs.*, **47**, 5086 (10 June 1953).

Single hairs suspended in loops in thioglycollate solution were stretched up to 10% by a load of 20 g., the time from the start of immersion to the end of stretching being recorded. One hundred hairs from each subject were used, and the results treated statistically. The stretch frequency pattern was directly related to the degree of swelling of the fibres as determined microscopically. Hair soaked in  $\text{NH}_4$  or Na thioglycollate soln. of various pH values showed a minimum at pH 5–6. At pH 9 Na thioglycollate showed a second minimum, while  $\text{NH}_4$  thioglycollate showed a maximum between pH 9 and 10. At pH 9.5 for each time of immersion the concentration of thioglycollate necessary to obtain an optimum frequency of stretching was reduced by adding salts, NaCl and  $\text{Na}_2\text{SO}_4$  being most, and KCN least, effective. After addition of salt to thioglycollate, hair damaged by bleaching gives the same stretching frequency pattern as the one obtained with normal hair from the same head in thioglycollate alone. The amount of salt necessary can be used as a measure of the damage caused by bleaching.

C. O. C.

**Further Studies on Digestion of Wool Keratin by Papain-Urea: Effect of adding Compounds that Rupture Disulphide Bonds in Alkaline Solution.** F. G. Lennox and H. M. Fors. *Australian J. Biol. Sci.*, **6**, 118–129 (1953); *Chem. Abs.*, **47**, 7219 (25 July 1953).

Optimum pH for digestion of wool by papain-bisulphite-urea is 7. Replacing bisulphite by Na thioglycollate, cysteine hydrochloride, or KCN makes high pH necessary for optimum digestion. Thioglycollate at pH 9.5 is better than cysteine at pH 9.5 or bisulphite at pH 7. KCN is less effective. In wool incubated in solutions of bisulphite, thioglycollate, cysteine, or KCN, in absence of papain and urea, splitting of disulphide bonds runs parallel with enzyme digestion in presence of these reagents. Digestion of wool in a solution containing only fully active papain and urea is 0.1% of that observed when bisulphite also is present. Collagen (which is free from cystine) is digested most rapidly in papain-thioglycollate-urea at pH < 7. At all pH values presence of both urea and a reagent splitting

the disulphide bonds leads to greater digestion in papain solutions than when either is present alone. The combined action of bisulphite and thioglycollate in papain-urea solution increases the digestion of wool over a range of pH values. C. O. C.

#### Development and Production of Synthetic Fibres in Europe. G. Abrahamson. *Chem. Eng. News*, 31, 2996-2997 (20 July 1953).

All the industrial European countries have, in spite of many difficulties, initiated plans for producing synthetic fibres, the U.K. planning to produce 30,500 short tons, Germany 10,000 tons, France 11,500 tons, and Italy 4000 tons.

Nylon is still the most important of these fibres, the largest producer being British Nylon Spinners Ltd. Imperial Chemical Industries Ltd. propose to produce 5000 tons of Terylene annually at Wilton, as well as Ardil at their Dumfries plant. British Celanese Ltd. are working on a fibre based on polyaminotriazines. In Germany 5000 tons of Perlon is being produced annually, but only Deutsche Rhodiaceta AG., of all the firms who have taken out licences, is actually producing nylon, annual output being 120 tons, to be raised to 600 tons; they also make Rhovyl. Cassella Farbwerke Mainkur have developed a cheap production process for polyacrylonitrile fibres, and Phrix-Werke AG., Hamburg, are to produce Redon, a fibre similar to Orlon. Phrix have also developed Philon, made from furfuraldehyde, but large-scale production has not been started as yet. In Italy the only fibre of which more than 1000 tons a year is made is nylon, but Perlon, Orlon, and Movil (Italian name for Rhovyl) also are being made, and erection of a Terylene plant is being considered. Nylon has been produced in Switzerland for the last two years, and production has now started of Mirlon (a Perlon-type fibre). Switzerland also produces Grilon (a polyamide fibre based on caprolactam) from imported intermediates, which are to be replaced by wood-hydrolysis by-products. In Holland Enkalon (a polyamide fibre) has been produced since early 1952, and production is soon to be doubled. Sweden is planning production of polyacrylonitrile fibres, and in Spain both polyamide and polyacrylonitrile fibres are to be produced at the rate of several thousand tons a year by Swiss processes. C. O. C.

#### Effect of Heat on the Length and X-Ray Pattern of Polyurethane Fibres. H. Zahn and U. Winter. *Kolloid-Z.*, 128, 142-153 (Oct. 1952).

Bristles of Perlon U (a polycondensate of 1:6-hexamethylene diisocyanate and 1:4-butyleneglycol) were cold-stretched up to 500% into long fibres. These fibres contract 1% when heated slack in dry air at 100°C. for 24 hr., the contraction increasing to 14% at 170°C., while the extension at break increases from the normal value (11%) to 16% at 100°C. and 33% at 170°C. When the fibres are held clamped in a stretching frame while being heated, small increases in length (<1%) occur, and the breaking extension becomes smaller, being 13% at 100°C. and 20% at 170°C. Heating slack fibres in water causes a 15% contraction at only 145°C., and the extension at break rises to 32%.

Under similar conditions the X-ray equatorial interferences become sharper, that of the 2-33-Å spacing being most affected, its line-width being halved. The diffuse ring at 4 Å., however, remains unchanged. This indicates improved side-by-side orientation of the chain molecules. The increased spread along the arcs, however, shows a definite disorientation of the crystallites. The 3-77-Å. and 4-20-Å. spacings are slightly reduced by dry heat to 3-67 Å. and 4-09 Å. respectively, but the 75-Å. spacing increases up to 114 Å. when the fibres are heated under constraint and to 120 Å. when heated slack; on heating slack fibres in water at 145°C. it lengthens to 132 Å.

Heating clamped fibres in 5% phenol solution at the higher temperatures reduces the original 7-3% contraction and 27-5% increase in diameter to a small elongation (<1%) and a mere 3% radial swelling; on heating slack at 170°C., there is a 5% increase in length and a 4-5% swelling.

After heating similar Perlon L (polycaprolactam) fibres in water the 63-68 Å. long spacing changes to 92-94 Å. There is also a remarkable reversible transformation of the long meridian period (114 Å.): on stretching the fibres 10% at 170°C. it increases to 140 Å., on release it returns to 120 Å. L. P.

#### PATENTS

##### Yarns for Bouclé or Similar Fabrics. American Viscose Corp. *USP* 2,621,392

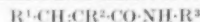
A single yarn of an organic ester of cellulose is doubled with a single yarn composed of fibres which are unaffected by aqueous solutions of ammonia or lower alkylamines. The doubled yarn is then treated with aqueous ammonia or alkylamine until it has no cellulose ester content, after which it is dried in absence of tension. C. O. C.

##### Super-stretching of Polyester Filaments, Films, etc. DuP. *BP* 695,567

Freshly melt-extruded, amorphous, polyethylene terephthalate filaments, etc. can be stretched up to 75 times their original length if stretching is carried out at 20-60°C. above the apparent minimum crystallisation temperature for the amorphous polyester. The stretched product has negligible molecular orientation or crystallisation, and can be subsequently drawn or otherwise treated to improve its tenacity or elongation. C. O. C.

##### N-Substituted Unsaturated Amide Polymers. Celanese Corp. of America. *BP* 694,911

Stable high-mol.wt. fibre-forming polymers that can be melt-spun at relatively high temperatures are obtained by polymerising N-substituted amides of formula—



( $R^1 = H, Alk., or Ar$ ;  $R^2 = H or CH_3$ ;  $R^3 = hydrocarbon attached to the N by a tertiary C atom or cycloalkyl attached to the N by a secondary C, and has preferably a high degree of molecular symmetry).$  C. O. C.

##### Fibres and Films of Acrylonitrile Polymer. American Viscose Corp. *USP* 2,622,003

Extrusion of a homogeneous mixture of polymer containing > 70% acrylonitrile and dimethylacetamide into 20-70% (by vol.) aqueous dimethylacetamide results in tough, extensible fibres which can be severely stretched without breaking. C. O. C.

#### VII—DESIZING; SCOURING; CARBONISING; BLEACHING

##### PATENTS

##### Washing Sheet Material. N. V. Philips' Gloeilampenfabrieken. *BP* 694,532

Speedy cleansing of cloth, paper, etc. is achieved by wetting the material with a dil. soln. of a non-ionic wetting agent and then causing the washing liquor to flow through the material at right angles to the surface of the sheet. C. O. C.

##### Operating a Dry-cleaning Machine. Wacker-Chemie. *BP* 696,232

Drying and/or ventilation of the cleansed material is effected by drawing hot air through the cleansing vessel and expelling it into the open, if desired through a condenser. This keeps the cleansing vessel under reduced pressure, so that there can be no leakage of solvent-laden air. C. O. C.

##### Decolorising or Bleaching of Ester Waxes. Promoters & Investors. *BP* 695,561

Caruba and other ester waxes are emulsified in presence of a chlorinated bleaching agent, e.g. NaOCl, and HCl is gradually added to liberate chlorine. C. O. C.

#### VIII—DYEING

##### Chemical Identification of Vegetable Dyes used on Ancient Japanese Silk (Preliminary Report).

K. Hayashi, T. Isaka, and G. Suzushino. *Misc. Rept. Research Inst. Nat. Resources*, No. 17-18, 33-42 (1950); *Chem. Abs.*, 47, 6660 (10 July 1953).

Examination of aqueous and alcoholic extracts of ancient Japanese silk fabrics resulted in identification of the following compounds and sources—Yellow dyes: berberine (mixed at times with copisine, worenine, and palmatine) from *Phellodendron amurense* var. *suberosum* and *Coptis japonica*, luteolin from *Miscanthus tinctorius* and *Arthaxon hispidus*, curcumin from *Curcuma dom-stica*, crocin glucoside from *Gardenia jasminoides*, rutin from *Sophora japonica*, and fustin from *Rhus trichocarpa*. Red dyes: pseudopurpurin from *Rubia akane*, alizarin from *Rubia tinctorum*, carthamin glucoside from *Carthamus tinctorius*, and brasilin from *Cesalpinia sappan*. Purple dye: shikonin from *Lithospermum erythrorhizon*. Blue

dyes: indigo from *Polygonum tinctorium*, *Mercurialis leucocarpa*, *Strobilanthes flaccidifolium*, and *Leatis sp.*; awobanin from *Commelina communis*. Brown and dark-brown dyes: tannins from various sources. C. O. C.

#### Protective Power of Surface-active Agents for Dyes.

R. Goto, T. Sugano, and N. Hayama. *Bull. Inst. Chem. Research, Kyoto Univ.*, **31**, 133-134 (1953); *Chem. Abs.*, **47**, 6663 (10 July 1953).

Work is described which indicates that the interaction between both ionic and non-ionic surface-active agents and dyes is not necessarily ionic. C. O. C.

**Standardizing the Colour of Butter.** A. Reinart and R. W. Brown. *Canadian Dairy Ice Cream J.*, **31**, (5), 31-33, 78 (1952); *Chem. Abs.*, **47**, 6568 (10 July 1953).

The hue of butter fat in cream is measured to enable that amount of colouring matter to be added to the cream which will give the butter a desired standard hue. The butter fat is separated from the cream by using the Minnesota reagent, the separated fat is dissolved in petrol, and its hue compared with known standards. C. O. C.

#### PATENTS

#### Dyeing with Vat and Sulphur Dyes.

G. BP 694,070  
The material is impregnated with an aqueous vat or sulphur dye composition, and then passed through a neutral, inert, water-immiscible oil which is not a solvent for the dye. The oil is at such temperature that steam is developed from the aqueous dye composition, the temperature of the oil and the duration of immersion being such that the material is not completely dried. On leaving the oil the material passes through squeeze rollers, after which it does not usually retain > 5% of its weight of oil. This residual oil is removed by passing the material through an aqueous bath containing a dispersing agent. Oxidation, soaping, etc. are then carried out in the normal way. C. O. C.

#### Dyeing with Vat Dyes at High Temperatures.

Amerienn Cyanamid Co. BP 693,251  
Cobalt and its compounds prevent decomposition and dulling of the dyeings when vat dyes are dyed at temperatures at which they are normally susceptible to over-reduction. C. O. C.

#### Dyeing or Printing Wool with Sulphuric Esters of Leuco Vat Dyes.

G. USP 2,618,529  
Stable solutions suitable for padding or printing animal fibres contain up to 20% of a sulphuric ester of a leuco vat dye, > 10% urea, 0.3-5.0% of a free acid to bring the pH to 1-3, no strong electrolyte, water, and, if desired, a thickening and/or wetting agent. Oxidation can be carried out as usual, and heavy dyeings or prints bleed neither into the developing bath nor on to white wool. If, during development with a dichromate, orthophosphoric acid is used instead of sulphuric acid, then rapid and complete development is obtained without overoxidation, the dyeings and prints being of excellent fastness. C. O. C.

#### Simultaneous Dyeing and Dimensional Stabilisation of Textile Fabrics.

American Viscose Corp. BP 693,497  
Fabrics composed predominantly of regenerated cellulose are simultaneously dyed and given dimensional stability by treating them as follows—(1) desize, scour, and dry; (2) simultaneously apply a vat dye and a special stabilising agent; (3) dry; (4) bake to set the stabilising agent; (5) reduce the vat dye, (6) reoxidise the vat dye; (7) scour; (8) dry; (9) tenter to desired width. The stabilising agent consists of a mixture of formaldehyde and a water-insoluble alkali-soluble cellulose ether. The liquor containing the dye and the stabilising agent contains also a strong mineral acid and a salt thereof, the pH being 1.2-6.5. The stabilising agent influences neither the reduction nor the re-oxidation of the dye. C. O. C.

#### Pigment-pad Dyeing of Cellulose Ester Fibres.

Celanese Corp. of America. USP 2,620,257  
Addition of a water-miscible organic solvent or swelling agent to the reducing bath enables the time of treatment to be considerably reduced. Better penetrated dyeings result. Preferably 10-50% by wt. of the bath consists of the solvent or swelling agent. C. O. C.

#### Colouring Cellulose Esters or Ethers.

BrC. BP 695,719  
A wide range of dyes containing acid groups can be fixed on cellulose acetate or the like if there is simultaneously present on the cellulose acetate both urea and a thiocyanate. C. O. C.

#### Colouring Cellulose Ester or Ether Textiles.

BrC. BP 696,135  
The material is impregnated with a diazo compound stabilised with an amine, an enolic azo-coupling component, an organic base volatile in steam, and a liquid which in presence of the organic base is a swelling agent for the fibre. The colour is then fixed by steaming. The liquid used is preferably a mixture of water and an organic liquid, e.g. an alcohol. Good colour value and fastness to rubbing and scouring are obtained. C. O. C.

#### Dyeing Blends of Organic Derivatives of Cellulose and Animal Fibres.

Celanese Corp. of America. BP 695,853  
The cellulose derivative in the blend is dyed, and then the whole treated with a titanous salt to remove any dye which has gone on to the animal fibres, after which the latter are dyed. C. O. C.

#### Polyazine Dyes (IV p. 386).

Printing, Coating, Lacquering, or Colouring Sheet Materials (IX below).

Pigmented Prints and Dyeings (IX below).

## IX—PRINTING

#### PATENTS

#### Printing on Cellulose Ester or Ether Textiles.

BrC. BP 696,134  
When thiocyanate is used to assist fixation during printing with acid or other dyes or dye intermediates, better results are obtained if the textile is first impregnated with the thiocyanate and then printed with a paste containing no thiocyanate. This avoids the weakening of the material caused when high concentrations of thiocyanate are present in the printing paste. C. O. C.

#### Printing, Coating, Lacquering, or Colouring Sheet Materials.

Bousley, French & Co. BP 693,281  
In printing or colouring where several colours are applied in succession, the printing units can be placed close to one another if drying of each colouring composition applied is carried out by a gas which is supplied to and withdrawn from an element having a working face which is close to and conforms to the shape of the surface to be dried. This working face is formed so as to force the drying gas to follow a path at variance with its natural flow path, which causes the surface to be dried to be continuously exposed directly to the gas. C. O. C.

#### Pigmented Prints and Dyeings.

Busf. BP 694,076  
Dyeings or prints which are very fast to washing and rubbing are produced by treating the material, before, during, or after application of a pigmented dispersion or solution of a high-mol.-wt. substance containing hydroxyl, carboxyl, or carbamoyl groups, with a polyisocyanate, and then baking the dried material above 100°C. C. O. C.

#### Washable Transfers.

H. E. Peace. BP 694,168  
A washable transfer suitable for application to cloth, leather, etc. consists of a thermosetting melamine resin, a plasticiser, a solvent, and a pigment. It is readily applied by means of a hot iron or a steam-heated pad, the resin being cured during application. C. O. C.

#### Printing on Plastic Resinous Articles during their Moulding.

Columbia Protokosote Co. USP 2,619,679

#### Single-stage Multicolour Printing.

Chromostyle. BP 695,712  
A roller or block is used which has several coloured elements made of a thermoplastic composition, printing being effected in presence of a solvent for the thermoplastic composition, which solvent, where the surface to be printed is also a thermoplastic, is also a solvent for the surface. C. O. C.

**Fast Diazo Prints.** Kalle & Co.

BP 695,824

2:3-Dihydroxynaphthalene-6-sulphonic acid and its salts are excellent coupling components for use with light-sensitive layers containing as the essential component a compound of formula—



( $\text{R}^1$  and  $\text{R}^2$  = same or different propyls which may be substituted by two  $\text{CH}_3$  or by one  $\text{C}_2\text{H}_5$  group). C. O. C.

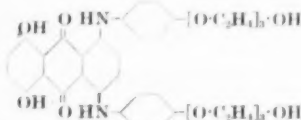
**Tinted Photographic Film Base.** Eastman Kodak Co.

USP 2,622,026

The film is tinted for antihalation purposes with a red dye having its maximum absorption in the blue and green regions of the spectrum and with a blue-green dye having its maximum absorption in the red and infrared. The red dyes preferably used have a nitrophenylazophenyl structure, e.g.—



and the blue-green dyes are dihydroxy bis(hydroxyalkoxyphenylamino)anthraquinones in which the hydroxyl and the hydroxyalkoxyphenylamino groups are in the 1, 4, 5, and 8 positions, e.g.—

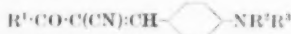


C. O. C.

**Dyes for Photographic Filters and the Discharge of such Dyes.** ICI.

BP 695,873

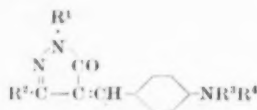
Dyes of formula—



( $\text{R}^1$  = alkaryl (Alk of  $> 4\text{C}$ ) which may be substituted by an arylamino group;  $\text{R}^2$  = H or subst. Alk;  $\text{R}^3$  = subst. or unsubst. Alk; the phenylene nucleus may be substituted) can replace colloidal silver in yellow filter layers and are free from the disadvantages associated with colloidal silver.

BP 695,874

Dyes of formula—



( $\text{R}^1$  and  $\text{R}^2$  = H, Alk, Ar, or alkaryl;  $\text{R}^3$  = subst. or unsubst. Alk;  $\text{R}^4$  = H or subst. Alk; the phenylene nucleus may be substituted; and either  $\text{R}^1$  or  $\text{R}^2$  is substituted by a group containing an alkyl chain of  $> 4\text{C}$ ) are used.

BP 695,875

The above dyes are discharged by treatment with an aq. soln. of a compound of formula  $\text{R-NH}_2$  ( $\text{R}$  = alkyl, alkaryl, amino, alkylamino, arylamino, aminohydroxy, or hydroxyl).

BP 695,876

A subst. or unsubst. arylenediamine is used as the discharging agent.

C. O. C.

**Multicolour Photography.** J. A. Ball.

BP 694,421

A four-component subtractive colour process. C. O. C.

**Multicoloured Photographic Prints.** Lawes Bros.

BP 694,580

Material bearing a light-sensitive compound is exposed to light through a transparency bearing a design, and then selected unexposed portions of the material are successively treated with different coupling components to produce a different colour in each.

C. O. C.

**Polyazine Dyes** (IV p. 386).

Dyeing or Printing Wool with Sulphuric Esters of Leuco Vat Dyes (VIII p. 389).

Colouring Cellulose Esters or Ethers (VIII p. 389).

Colouring Cellulose Ester or Ether Textiles (VIII p. 389).

**X—SIZING AND FINISHING****Adsorption of Dyes by Montmorillonite.** B. S. Emodi.

*Clay Minerals Bull.*, 3, 76-77 (1949); *Chem. Abs.*, 47, 6215 (10 July 1953).

Strong aq. soln. of various basic dyes were shaken with small weighed quantities of fairly pure Ca montmorillonite, the dye being always in large excess over the clay. After shaking, the mixtures were filtered, and the amounts of Ca and dye in the filtrate determined. The so-called ionic adsorption of dyes is not a single base-exchange reaction but results in excess Ca ions being left in the clay structure. This may be due to the dye molecule being arranged between the Si surfaces around the edges of the clay flakes and hindering removal of Ca ions.

C. O. C.

**Dimensional Stabilisation of Cellulose Fabrics.**

Fothergill &amp; Harvey.

BP 696,282

Fabric of natural and/or regenerated cellulose is treated in presence of aqueous caustic alkali, and preferably an alkali-metal salt as an accelerator, with an aliphatic or alicyclic compound containing at least two halogen atoms, at least two of which are not on the same C atom and are capable of forming epoxide rings with at least one OH group, which also must be present in the molecule; or alternatively, the compound must contain at least one halogen atom and at least one epoxide ring, which, if not adjacent to a halogen atom, must be accompanied by an OH group capable of forming a second epoxide ring with at least one halogen atom. Suitable compounds include 1:3-dichloro-2-hydroxypropane and epichlorohydrin. It is believed that the reaction causes cross-linkages of the type  $-\text{O}-\text{CH}_2-\text{CH}(\text{OH})-\text{CH}_2-\text{O}-$  to be formed at intervals of about 30 glucose units.

C. O. C.

**Crease-resisting Cellulosic Textiles.** Tootal Broadhurst

Lee Co.

BP 695,703

The textile is impregnated with an aqueous solution of a crystalloidal synthetic resin precondensate and with an aqueous dispersion of a polysiloxane. The polysiloxane must be an oily liquid, and on being heated in thin layers must yield an infusible non-tacky flexible film. The impregnated textile is then baked. Impregnation may be carried out either in two steps or in one operation.

C. O. C.

**Permanent Finish on Cellulosic Textiles.** Cluett,

Peabody &amp; Co.

BP 693,111

A wide variety of permanent finishes are produced on cellulosic textiles without much alteration in handle or porosity by first modifying the cellulose topochemically to an unsaturated ester or ether without altering its physical form, and then copolymerising it with an olefinic monomer. The topochemical treatment should be such that the product contains one unsaturated substituent per 1-10 anhydroglucose rings.

C. O. C.

**Making Mechanical Finishes on Cellulose or Silk resistant to Washing.** Bradford Dyers' Assn.

BP 696,029-30

Mechanical finishes on natural or regenerated cellulose or silk, e.g. glazing, moiré, embossing, or compressive shrinking, are rendered resistant to washing by treating the material before, during, or after application of the mechanical finish with an organic isocyanate or isothiocyanate in the form of either vapour, aqueous dispersion, or organic-solvent solution, and then heating to cause the isocyanate or isothiocyanate to react with the fibre. If the reaction is carried out in the presence of an amine, amidine, or aminotriazine, firmer handle and even better resistance to washing are obtained.

C. O. C.

**Flameproof Cellulose Derivatives.** Celanese Corp. of America.

BP 694,217

Organic derivatives of cellulose containing free hydroxyl groups can be flameproofed and in some cases even rendered completely and permanently non-flammable by esterifying them with a halogenoalkyl halogenophosphonate, e.g. bis-2-chloroethyl chlorophosphonate. The flameproof finish produced is fast to washing and dry cleaning.

C. O. C.

### Treating Flexible Materials with Powder.

**Tootal Broadhurst Lee Co.** BP 693,619  
The material, e.g. cloth, is passed through the apparatus twice. The first time through, powder is applied to it; and the second time through, it is caused to deviate from the path it followed on the first occasion and pass through a bight where the powder is largely removed, e.g. by beating and/or suction and/or treatment with a liquid, and then return to the first path. This method is especially suitable for applying bleaching powder to wool fabrics to reduce their tendency to felt. C. O. C.

### Combined Shrink-resisting and Mothproof Finish on Textiles containing Wool.

**American Cyanamid Co.** USP 2,620,322  
Textiles containing wool are impregnated with an aqueous solution containing an alkylated hydroxymethylmelamine and triethanolamine silicofluoride, and then dried and baked. On washing the treated material is dimensionally stable and the mothproof effect is not impaired. C. O. C.

### Producing a Suède Finish on Cloth.

**D. E. Mulholland.** USP 2,617,170  
The cloth is kept under constant tension both warp- and weft-ways while being treated with an abrasive roller, means being provided to dissipate or carry away the heat evolved by the friction between the abrasive roller and the cloth. C. O. C.

### Laundry Starch.

**Universal Oil Products Co.** USP 2,620,281  
Garments treated with a starch composition containing a water-soluble or water-dispersible antioxidant for oils, e.g. a lower alkoxyphenol or lower alkylalkoxyphenol, are more readily cleansed than goods which have been starched in absence of an antioxidant. C. O. C.

Further Studies on Digestion of Wool Keratin by Papain-Urea: Effect of adding Compounds that Rupture Disulphide Bonds in Alkaline Solution (VI p. 387).  
Simultaneous Dyeing and Dimensional Stabilisation of Textile Fabrics (VIII p. 389).

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

**Paper of improved Wet Strength.** DuP. BP 695,148  
Paper of improved wet strength is made by adding to the paper pulp 0.5–25% (or 0.5–3%) based on the dry weight of the paper, of a urea-formaldehyde condensate modified with hexamethylenediamine. S. V. S.

### Coating Composition for Paper.

**American Cyanamid Co.** USP 2,620,316  
Addition of a small amount of a water-soluble amino resin to coating composition containing soya protein imparts improved water resistance to the coated paper. C. O. C.

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

### Compositions for Pickling Animal Hides and Skins.

**Diamond Alkali Co.** BP 694,614  
When a mixture of alkali-metal dichromate crystals and sulphuric acid is fused, the product obtained from the upper layer formed is used together with an alkali-metal chloride or the by-product sodium sulphate obtained when sodium dichromate is crystallised from acid chromate liquid; when used for pickling hides, this results in leathers of better handle than those pickled in the ordinary way. C. O. C.

### Tanning.

**W. Fabri.** BP 694,213  
Tanning, rinsing, oiling, and neutralising can be successively carried out in the same vessel if the deaired hides are stretched on frames which are grouped in parallel vertical planes in the tank. These frames are stationary, the liquids being circulated between the hides either by being pumped or by the introduction of compressed air. C. O. C.

### Tanning with a Sulphonate Tanning Agent and a Formaldehyde-Arylamine-Cyanamide Condensate.

**American Cyanamid Co.** USP 2,621,102  
Leather of excellent plumpness, fullness, fine smooth grain, and good handle is obtained by use of a sulphonate

tanning agent and a compound of formula  $R\{CXY-R\}^n$  ( $R$  = monoguanidine or monodiguanide-substituted arylene radical;  $n$  = a whole number;  $X$  and  $Y$  = H or hydrocarbon radicals of 1–3 C). C. O. C.

### Making Leather resistant to Water Penetration.

**G. H. von Fuchs.** BP 694,586  
Leather is made resistant to penetration by water by incorporating into the leather a monomeric acid-reacting succinic acid derivative of  $>17$  C., e.g. *isooctadecenylsuccinic acid*. C. O. C.

## XIII—RUBBER; RESINS; PLASTICS

### Polyesters from Eicosane-1:20-diol and Dicarboxylic Acids.

**V. V. Korshak and S. V. Vinogradova.** Doklady Akad. Nauk S.S.S.R., 89, 1017–1020 (21 April 1953).  
Polyesters are prepared by condensation of eicosane-1:20-diol with each of the first nine members of the series of saturated straight-chain dicarboxylic acids. Their m.p., which vary in periodic fashion with the number of carbon atoms in the acid, remain almost const. at 85–88°C. when this number is even, and rise from 67° to 84°C. with increasing chain length when the number is odd. A. E. S.

### Solubilisation of Polyvinyl Acetate.

**N. Sato and S. Saito.** Kolloid-Z., 128, 154–158 (Oct. 1952).  
Polyvinyl acetate (P.V.A.) by itself is insoluble in water, but dissolves in sufficiently concentrated sodium dodecyl sulphate to give a completely clear and stable solution. The higher the degree of polymerisation, the more solubiliser is needed. The viscosity of P.V.A. in such solutions is greater than in acetone. Thus the P.V.A. molecule is more mobile in soap solution than in the so-called good solvent acetone. The current micelle theory of solubilisation is incapable of explaining the above phenomena; the authors suggest that a new mechanism must be found, and put forward several that are possible. L. P.

### Molecular Weight and Intrinsic Viscosity of Polydimethylsiloxane.

**A. Ya. Korolev, K. A. Andrianov, L. S. Utesheva, and T. E. Vvedenskaya.** Doklady Akad. Nauk S.S.S.R., 89, 65–68 (1 March 1953).  
Osmotic pressure and viscosity measurements are made on toluene solutions of the products of the fractional pptn. by methanol of a benzene soln. of polydimethylsiloxane. The first fraction (14.3% yield) has an average mol. wt.  $M$  (from osmotic pressure) of 1,290,000, and is an elastic solid; the second and third fractions (12.5%, 30.6%;  $M$  = 408,000, 144,000) are plastic solids; and the fourth and fifth fractions (15.4%, 24.3%;  $M$  = 57,000, 24,000) are viscous liquids. The relation between intrinsic viscosity and  $M$  is far from linear ( $[\eta] \propto M^{0.65}$ ), indicating a high degree of flexibility in the polymer molecule. Also, the value of the const.  $K$  in the relation  $\eta_{sp} = [\eta]c + K[\eta]^2c^2$  increases with increase in  $M$ , which is considered to indicate, following J. Harris (*J. Polymer Sci.*, 8, 353 (1952)), that chain-branching occurs to a greater extent in the lower polymers. A. E. S.

### PATENTS

### Coloured Figured Thermoplastic Artificial Materials.

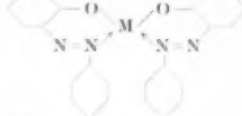
**Lonza Elektrizitätswerke & Chemische Fabrik n.** BP 695,598

The thermoplastic material, mixed with the plasticiser and, if necessary, filling material, before gelatinising, is monochromatically dyed, and heated until it becomes possible to crumble it but it is not gelatinised. The product is then crushed and mixed with material of a different colour, and the mixture heated until it is gelatinised. A multicoloured product is obtained without any bleeding of the two colours into each other. C. O. C.

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Coating Composition (V p. 387).

### XIV—ANALYSIS; TESTING; APPARATUS

#### Determination of Aldehydes using the Reaction with Primary Amines. S. Siggia and E. Segal. *Anal. Chem.*, **25**, 830-831 (May 1953).

In the reaction—



there is a definite equilibrium; the reaction has thus to be carried out in a non-aqueous medium (ethylene glycol-2-propanol). The excess amine unused in the reaction is titrated with salicylic acid in the same solvent, this being insufficiently strong to hydrolyse the Schiff's base formed in the forward reaction. Acids which are stronger than salicylic acid bind the amine and thus interfere. Aromatic amines are too weak to be effectively titrated by salicylic acid, and laurylamine is used as reagent; it has the advantage of low volatility. With the exception of H-CHO, aliphatic aldehydes do not react. The method, together with examples of its application, is given. J. W. D.

#### New Colour Reagent for Determination of Hexoses. B. Klein and M. Weissman. *Anal. Chem.*, **25**, 771-774 (May 1953).

A violet colour is produced by the action of a chromotropic acid soln. in 15 M-H<sub>2</sub>SO<sub>4</sub> upon hexoses, and it may be used to determine them in the presence of pentoses. Many disaccharides, such as lactose, maltose, and sucrose, also react with the reagent. Hexoses are converted to 5-hydroxymethylfurfuraldehyde, the hydroxymethyl group being split to form HCHO, which reacts with the chromotropic acid. Under the same conditions, pentoses yield furfuraldehyde, which does not produce HCHO, and thus they do not affect the reaction with hexoses. The resulting colour intensity is related linearly to the concn. of hexose used, but the yield of HCHO is not quantitative, probably because the fission which leads to its formation is one alternative path for the decomp. of hexoses in strong acid. The test procedure is described, and the theory of the reactions involved is discussed at length. The course of certain of the reactions has been studied by the use of isotope-labelled compounds. J. W. D.

#### Specific Colour Reaction for Sugars. H. Tauber. *Anal. Chem.*, **25**, 826 (May 1953).

A 0.2% soln. of the sugar is caused to react with a soln. of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in conc. H<sub>2</sub>SO<sub>4</sub>, in the presence of a soln. of aminoguanidine; colour development takes place within 1 min. An intense blue colour is given by carbohydrates which contain only aldohexose units (glucose, mannose, glycogen, starch, dextrin, cellulose, etc.); a wine-red colour by those containing ketohexose units (fructose, raffinose, inulin, etc.); and a yellow colour by pentoses (arabinose, xylose, etc.). The control is colourless. J. W. D.

#### Amperometric Method for Determining the Sorption of Iodine by Starch. B. L. Larson, K. A. Gilles, and R. Jenness. *Anal. Chem.*, **25**, 802-804 (May 1953).

The activity of iodine in a starch-iodine soln. is chiefly a function of the amount and the physical condition of the amylose present in the starch. The equilibria in such systems have been investigated by titrating with KIO<sub>3</sub> acid soln. containing starch and KI; the curves for current flow versus KIO<sub>3</sub> added have been plotted for soln. of purified amylose and amylopectin. The plot for the reagent blank shows a linear relationship between current (over the range 0-10 μA) and the free iodine present. Curves obtained in the presence of amylose and amylopectin show points of inflection, and it is shown how, by extrapolation, they may be used to estimate the iodine which is specifically bound by the amylose helices, and that which is non-specifically bound by both amylose and amylopectin, this latter being greater in the case of amylopectin. The small initial sorption of iodine by amylopectin is attributed to the presence of some amylose as impurity. The iodine binding of amylose is shown to be substantially independent of amylose concn. over a wide range. Amylopectin does

not interfere in the titration of amylose in the region of the end-point, and no interference was observed with several pentosans which were studied in this connection. The total iodine-binding capacity and calc. amylose contents of a number of starches agree well with accepted values obtained potentiometrically. The method, the advantages of which are discussed, is given fully. J. W. D.

#### Reaction of Naphthaquinone-4-sulphonate with Imino Acids. W. Troll. *J. Biol. Chem.*, **202**, 479-485 (May 1953).

Imino acids form mononaphthaquinone derivatives quantitatively with 1:2-naphthaquinone-4-sulphonate at room temperature, a fivefold excess of naphthaquinone-sulphonate over the imino acid being used in a bicarbonate buffer (pH 8.3); the solution is acidified and the product crystallised out. A method for the determination of imino acids in the presence of amino acids in protein hydrolysates has been developed. P. G. M.

#### PATENTS

#### Device for Use in Formulating Dyeing Recipes.

Nederlandse Organisatie voor Toegepast-Natuurwetenschappelijk Onderzoek ten Behoeve van Nijverheid, Handel en Verkeer. BP 693,356

Each sheet of three sets of translucent superimposable sheets carries a graph. In one of the sets the graph has the X-stimulus of the eye as its parameter, similarly another set has the Y-stimulus, and the third set the Z-stimulus, the X-, Y-, and Z-stimuli being those defined by the International Commission for Illumination. Each graph comprises a set of curves representing for a given value of one parameter, and independently of the other two parameters, the possible combinations between three chosen dyes to produce, on a specified material under specified dyeing conditions, the value of the parameter. The curves are plotted in a system of co-ordinates, one axis representing the concentration of one dye, the other axis that of the second dye, and each curve representing a given concentration value of the third dye. A dyeing recipe for a given sample can then be formulated by (1) determining the X-, Y-, and Z-stimuli of the sample to be matched, (2) selecting three charts having the same parameter values as these stimuli, and (3) finding the intersection point of the curves on these charts, which gives the common concentration point for the three dyes needed to match the sample. C. O. C.

#### Determining Daylight Fluorescence. IC. USP 2,620,445

A beam of monochromatic or a narrow waveband of light is directed into the sample, and the light emerging from the sample is examined to see whether or not visible radiation of other than the incident spectral quality and in addition thereto is being emitted from the specimen. C. O. C.

#### Measuring Moisture in Material (I p. 382).

#### Measuring or Controlling Humidity (I p. 382).

#### Nomogram for Ammonium Sulphate Solutions (III p. 383).

#### Standardising the Colour of Butter (VIII p. 389).

### XV—MISCELLANEOUS

#### Adsorption in Capillaries. C. Pierce and R. N. Smith. *J. Phys. Chem.*, **57**, 64-68 (1953); *Chem. Abs.*, **47**, 5211 (10 June 1953).

A review of the properties of porous solids. Capillaries are classified in two categories differing by the way in which adsorption occurs. The methods and limitations of pore-size calculations are discussed, as well as the importance of surface mobility and external capillaries in the adsorption process. C. O. C.

#### PATENT

#### Reducing Cuprous Oxide Coatings to produce Patterns or Designs. Kansas City Testing Laboratory. USP 2,616,841

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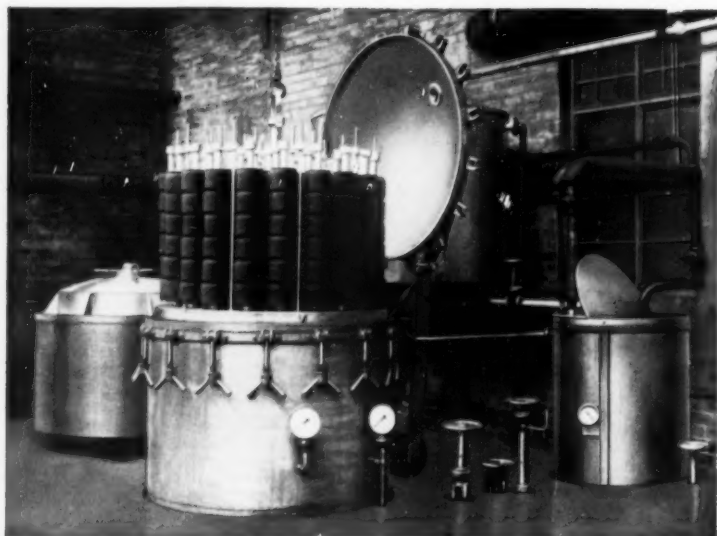
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## FORTHCOMING MEETINGS OF THE SOCIETY — continued from page x

## MANCHESTER JUNIOR BRANCH

All lectures to be held in the Reynold's Hall, Manchester College of Technology, at 6.30 p.m.

1953

Monday Dr. I. M. S. Walls. *Azoic Dyestuffs on Acetate Rayon*

Wednesday J. S. Ward, Esq., B.Sc. *Recent Developments in the Dyeing of Synthetic Fibres*

1954

Monday C. L. Bird, Esq. *Theoretical Aspects of the Dyeing of Cellulose Acetate Rayon*

Monday Lecture, title to be announced later

8th March

## HUDDERSFIELD SECTION

All lectures at Field's Cafe, Westgate, Huddersfield, at 7.30 p.m. unless otherwise stated

1953

Tuesday Sound Film, *Perspex and Polyvinyl Chloride Manufacture and Use*, with Lecture by J. H. Davies, Esq. (I.C.I. Plastics Division)

Friday ANNUAL DINNER. Princess Cafe, Huddersfield

20th Nov.

Monday  
23rd Nov.

J. L. Beadle, Esq. (Brown & Forth Ltd.).  
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Joint with Huddersfield Textile Society, to be held in Large Hall, Huddersfield Technical College

Tuesday  
8th Dec.

Dr. Baines. *Photography as a Scientific Implement*  
Joint with Huddersfield Section of Royal Institute of Chemistry

Tuesday  
15th Dec.

I. D. Rattee, Esq. (I.C.I. Ltd.). *Level Dyeing of Wool with Afterchrome Dyes*

1954

Tuesday  
19th Jan.

F. Thies, Esq. (Messrs. B. Thies, Coesfeld). *High Temperature Dyeing and Pressure Drying*

Tuesday  
16th Feb.

J. G. Grundy, Esq. (Clayton Aniline Co. Ltd.). *Cibalan Dyes and their Fastness Properties on Nylon and Application to Wool-Nylon Unions*

Tuesday  
16th Mar.

ANNUAL GENERAL MEETING, followed by a lecture, *Fastness Properties for Users Satisfaction*. J. S. Ingham, Esq. (Marks & Spencer Ltd., London)

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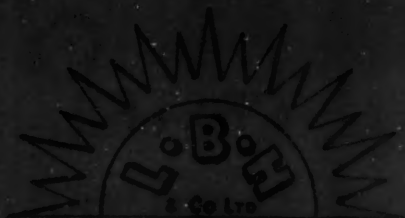
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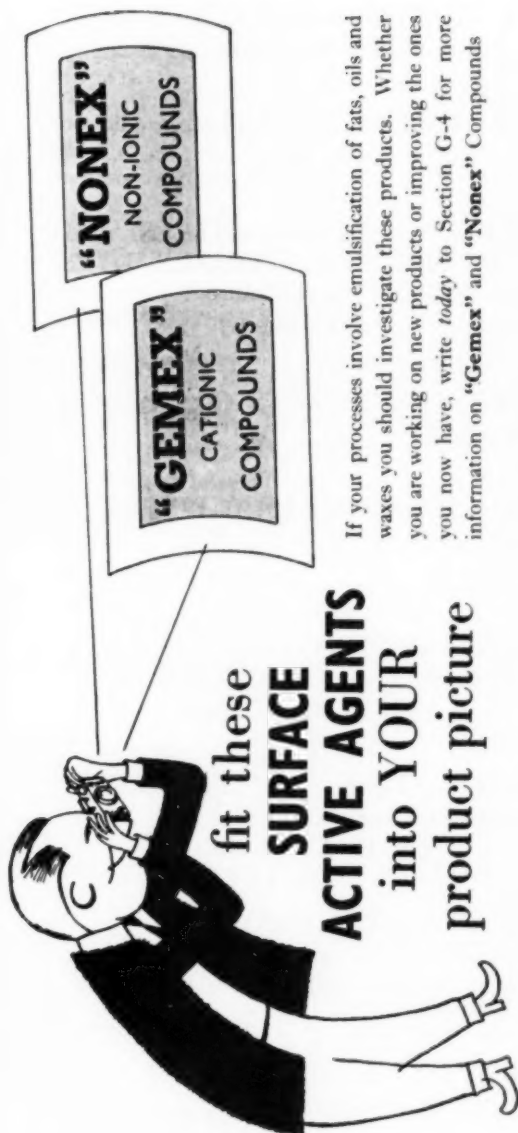
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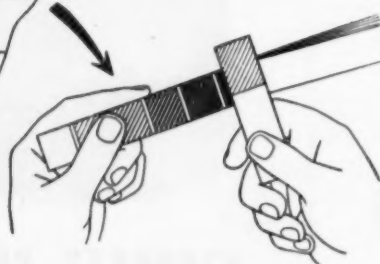
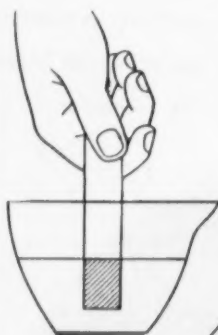
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